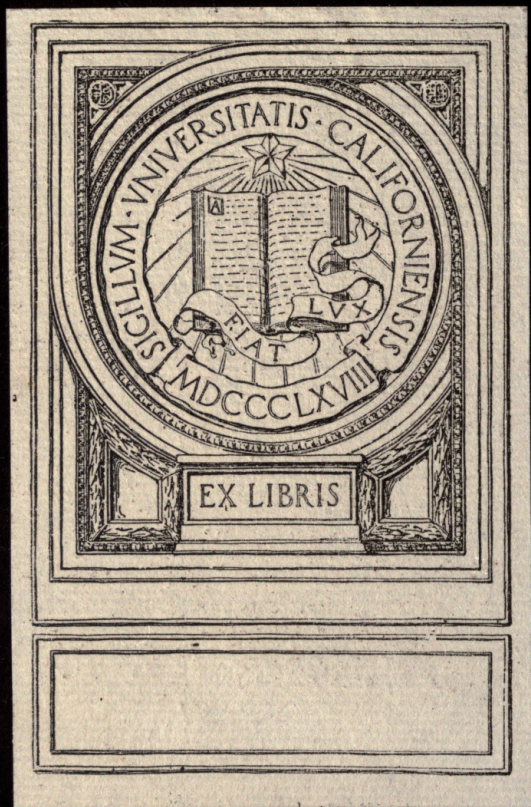


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# LABORATORY MANUAL

OF

# BITUMINOUS MATERIALS

FOR THE USE OF

## STUDENTS IN HIGHWAY ENGINEERING

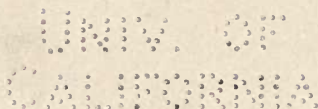
BY

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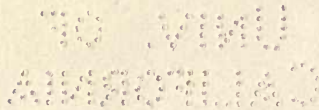


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## PREFACE

DURING the past five years a number of our leading universities have inaugurated courses in highway engineering in which laboratory instruction is given in the testing of bituminous materials. A general tendency appears to exist upon the part of many other educational institutions to develop the same line of activity because of the desire of highway engineers throughout the country to familiarize themselves with these materials.

While numerous technical and scientific papers have been published upon this subject in more or less detail, no attempt has as yet been made to furnish the student in highway engineering with a complete laboratory manual. As the result of his experience since 1912 in laboratory instruction in bituminous materials given in the Graduate Course in Highway Engineering at Columbia University, the author has endeavored to prepare a manual which may perhaps fill a want both upon the part of instructors and students and also those highway engineers in charge of State, county, or municipal work who have, or expect to have, a laboratory at their disposal. The author fully realizes that no first attempt along this line is apt to meet the many-sided demand, and will welcome criticisms and suggestions from those who are sufficiently interested to make them.

In the preparation of this manual no attempt has been made to trace down the obscure origin of many methods of testing which are described. The author has, however, endeavored to give credit in all cases where the originator is known, and if by chance any are unintentionally slighted, he takes this occasion to offer his sincere apologies. Many of the methods described are taken practically verbatim from U. S. Department of Agriculture Bulletin No. 314, by Hubbard and Reeve. Other publications which have been drawn upon for

original descriptions are "The Modern Asphalt Pavement," by Clifford Richardson; "Methods for Testing Coal Tars and Refined Tars and Oils and Pitches Derived Therefrom," by S. R. Church, and The Proceedings of The American Society for Testing Materials, and The American Society of Civil Engineers. For many helpful suggestions relative to the arrangement and development of the subject-matter of this book the author wishes to express his indebtedness to Professor Arthur H. Blanchard.

PRÉVOST HUBBARD.

WASHINGTON, D. C.  
July 15, 1916.



## INTRODUCTION

THIS manual is primarily intended as a laboratory guide for the student of highway engineering and not as a treatise upon methods of analyses and tests of bituminous materials. Its object is to describe methods in sufficient detail to enable the student to perform the more common and widely used tests with a reasonable degree of accuracy, provided his work is carefully conducted and he is supplied with the necessary laboratory equipment. Certain tests not in general use have been entirely eliminated from consideration, while others which have been included, together with those of minor importance, are shown in fine print to separate them from the more common and more important methods.

The arrangement of subject-matter in the following pages is designed to develop not only the abstract technique of the bituminous material laboratory, but also to deal with the interpretation of results of tests in connection with the identification, suitability for specific purposes, and control of uniformity of materials tested. The treatment is intended to be as brief as lucidity permits. Certain fundamentals not strictly a part of laboratory work have, however, been included in Part I for a guide to students not well versed in the nomenclature, classification, and uses of bituminous materials.

In the matter of interpreting the results of tests it should be realized that it is impossible to take into account all conceivable combinations of conditions. Moreover, our knowledge of the subject is yet far from being perfect, and many conflicting ideas are held by different highway engineers and chemists regarding certain matters. To some extent, therefore, interpretation of test results should be considered as opinions based upon personal observations at the time such opinions are expressed. As new facts are developed and observations are

extended, these opinions become subject to modification or entire change. If the matter is viewed in this light it becomes apparent that at the present time any dissertation upon interpretation of results should be considered more as a guide than as a collection of definite rules or laws which are not subject to modification.

There are a number of important considerations which have to be reckoned as variables affecting conclusions drawn from a comparison of laboratory tests with service results. The first of these is variations in test results from different laboratories, due to lack of standardization of methods, and also to careless operation. Too careful attention cannot be paid to even the apparently minor details of a method if consistent results are to be expected. The second point is, what constitutes satisfactory service results? That which is quite satisfactory to one engineer in view of past experiences may prove far from satisfactory to another of a more exacting nature. It is of course highly desirable to approach the ideal as nearly as possible, but in attempting this the question of what is practical under ordinary conditions must not be disregarded.

Quite frequently unsatisfactory service results are obtained from the improper or careless use of first-class materials. In such cases, unless the true cause of failure is plainly apparent, there is a natural tendency to place the blame upon the character of the material used. This is apt to lead to much confusion in interpreting the results of laboratory tests.

In the case of bituminous highways it should be remembered that the bituminous material constitutes but a relatively small proportion of the road structure, and that failure is quite as likely to be due to the use of inferior products other than the bituminous material. The quality of such materials as broken stone, gravel, sand, filler, wood, etc., is of as much importance from the standpoint of success or failure as of the bitumen. Moreover, some types and grades of bituminous materials which will prove satisfactory with one type of aggregate will not prove as satisfactory with another type of an apparently equivalent grading.

Another frequent cause of unsatisfactory service results is faulty construction. Thus, poor drainage may cause rapid disintegration and failure of a carefully constructed wearing course laid with good material. A rough, uneven, or poorly compacted foundation will almost invariably produce an unsatisfactory surface. Last but not least, the selection of the wrong type of pavement to meet existing traffic and other local conditions may result in failure through no inherent fault of the materials themselves except that they were not of the proper grade or character to meet the given conditions.

The laboratory may be made a valuable asset in highway engineering, but unless the engineer understands the principles involved in the analyses of bituminous materials and thoroughly appreciates the necessity of correlating test values with service results, it may prove to be a handicap, particularly in the matter of formulating specifications. There should be close cooperation and frequent interchange of ideas between the highway engineer and the testing engineer or chemist if the maximum value of the laboratory is to be developed.





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# LABORATORY MANUAL OF BITUMINOUS MATERIALS

FOR USE OF STUDENTS IN HIGHWAY ENGINEERING

## PART I—GENERAL

### USE OF THE MANUAL

Before attempting work in the bituminous material laboratory, the student who uses this manual should thoroughly familiarize himself with the subject-matter of Part I. When handling an unknown material he should first identify it as to type and grade. In most cases the general appearance of the material, together with its odor when warm and its apparent consistency, will enable him to do this. If still in doubt, a determination of specific gravity and a consistency test as described in Part II may be made. The interpretation of results under the specific gravity test may then be used to identify roughly the material under one of the following classes:

1. Fluid Petroleum Products and Emulsions.
2. Semisolid and Solid Petroleum and Asphalt Products.
3. Refined Tars and Tar Pitches.
4. Creosoting Oils.
5. Bituminous Aggregates.

When this is done, the student should turn to Part III. Under the proper classification he will find a list of the ordinary tests to make upon the material, together with comments upon the interpretation of results. These tests should then be made in accordance with methods described in Part II. The blank pages in Part III may be used to insert analyses of particular interest and additional facts which may develop relative to the significance of tests. After the student has thoroughly familiarized himself with the various types and



grades of bituminous materials, he will be prepared for more advanced work in connection with the specification of materials. It should be borne in mind, however, that the matter of preparing specifications should not be attempted until the student has had considerable field experience or has had an opportunity to observe the behavior of the various materials under known service conditions.

### IMPORTANT DEFINITIONS

The following definitions are considered important in connection with the use of this manual. For this purpose the terms are arranged in groups, according to logical sequence rather than alphabetically. Those marked with an \* have been adopted as standard by The American Society for Testing Materials; those marked with a † have been recommended to The American Society for Testing Materials by its Committee on Standard Tests for Road Materials, and those marked with a ‡ have been proposed to The American Society of Civil Engineers by The Special Committee on Materials for Road Construction and on Standards for Their Tests and Use. In certain instances comments have been added by the author and definitions have been included which have not been recommended to or adopted by any technical society.

#### DEFINITIONS OF MATERIALS

*Bitumens.\* †*—Mixtures of native or pyrogenous hydrocarbons and their non-metallic derivatives, which may be gases, liquids, viscous liquids, or solids, and which are soluble in carbon disulphide. Note: Broadly this term includes petroleums, asphalts, and tars which may or may not contain impurities insoluble in carbon disulphide. From a rigid interpretation of the definition such substances should be more properly termed bituminous materials unless free from impurities which are not bitumen.

*Bituminous.\**—Containing bitumen or constituting the source of bitumen. Note: (1) As bituminous aggregate. (2) As bituminous coal.

*Bituminous Material.*‡—Material containing bitumen as an essential constituent.

*Solid Bituminous Materials.\** ‡—Those having a penetration at 25° C. (77° F.), under a load of 100 grams applied for 5 seconds, of not more than 10.

*Liquid Bituminous Materials.\** ‡—Those having a penetration at 25° C. (77° F.), under a load of 50 grams applied for 1 second, of more than 350.

*Semisolid Bituminous Materials.\** ‡—Those having a penetration at 25° C. (77° F.), under a load of 100 grams applied for 5 seconds, of more than 10, and a penetration at 25° C. (77° F.), under a load of 50 grams applied for 1 second, of not more than 350.

*Bituminous Aggregate.*—A mineral or other aggregate containing bitumen as a cementing medium.

*Bituminous Mastic.*—A bituminous aggregate, the mineral portion of which consists of very fine particles.

*Bituminous Rock.*—Rock naturally impregnated with petroleum or asphalt.

*Bituminous Emulsion.*†—A liquid mixture in which minute globules of bitumen are held in suspension in water or a watery solution.

*Petroleum.*†—Liquid bitumen occurring as such in nature.

*Topped Petroleum.*†—Petroleum deprived of its more volatile constituents.

*Residual Petroleum.*—A liquid residue obtained by distilling petroleum to a point beyond which water and oils accompanying the elimination of water are removed.

*Blown Petroleum.\** ‡—Semisolid or solid products produced primarily by the action of air upon liquid native bitumens which are heated during the blowing process.

*Asphaltic Petroleum.*—Petroleum which, upon evaporation or fractional distillation without blowing, will yield an asphalt cement.

*Paraffin Petroleum.*—Petroleum which, upon evaporation or fractional distillation, will yield a greasy residue containing an appreciable quantity of paraffin hydrocarbons.



*Malthas*.—Very viscous petroleum.

*Asphalts*.\* ‡—Solid or semisolid native bitumens, solid or semisolid bitumens obtained by refining petroleum, or solid or semisolid bitumens which are combinations of the bitumens mentioned with petroleum or derivatives thereof which melt upon the application of heat and which consist of a mixture of hydrocarbons and their derivatives of complex structure, largely cyclic and bridge compounds.

*Native Asphalt*.\*—Asphalt occurring as such in nature.

*Refined Asphalt*.—Asphalt which has been subjected to a refining process but which is ordinarily too hard for use in the manufacture of bituminous pavements until softened by combining it with a flux. Note: Commonly designated in paving work as "R. A."

*Oil Asphalt*.—Asphalt manufactured directly from petroleum.

*Asphalt Cement*.\*—A fluxed or unfluxed asphalt especially prepared as to quality and consistency for direct use in the manufacture of bituminous pavements, and having a penetration at 25° C. (77° F.), of between 5 and 250, under a load of 100 grams applied for 5 seconds. Note: Commonly designated in paving work as "A. C."

*Rock Asphalt*.†—Sandstone or limestone naturally impregnated with asphalt.

*Asphalt Block*.—Paving block composed of compressed asphaltic concrete.

*Tars*.\* ‡—Bitumens which yield pitches upon fractional distillation and which are produced as distillates by the destructive distillation of bitumens, pyrobitumens, or other organic materials.

*Dehydrated Tars*.\* ‡—Tars from which all water has been removed.

*Refined Tar*.\* ‡—Tar freed from water by evaporation or distillation until the residue is of desired consistency, or a product produced by fluxing tar residuum with tar distillate.

*Coal Tar*.\* ‡—The mixture of hydrocarbon distillates, mostly unsaturated ring compounds, produced in the destructive distillation of coal.

*Gas-House Coal Tar*.\* ‡—Coal tar produced in gas-house



retorts in the manufacture of illuminating gas from bituminous coal.

*Coke-Oven Tar.\* ‡*—Coal tar produced in by-product coke ovens in the manufacture of coke from bituminous coal.

*Oil-Gas Tars.\**—Tars produced by cracking oil vapors at high temperatures in the manufacture of oil gas.

*Water-Gas Tars.\**—Tars produced by cracking oil vapors at high temperatures in the manufacture of carburetted water gas.

*Flux.\* ‡*—Bitumens, generally liquid, used in combination with harder bitumens for the purpose of softening the latter.

*Cut-Back Products.\**—Petroleum or tar residuums which have been fluxed with distillates.

*Pitches.\* ‡*—Solid residues produced in the evaporation or distillation of bitumens, the term being usually applied to residues obtained from tars.

*Straight-Run Pitch.\**—A pitch run to the consistency desired, in the initial process of distillation, without subsequent fluxing.

*Dead Oils.\* ‡*—Oils with a density greater than water which are distilled from tars.

*Creosoting Oil.*—Tar distillates, tars and mixtures of tars with tar distillates which are used by a process of impregnation in the preservation of wood. Note: This term was originally confined to the heavier coal-tar distillates carrying a large proportion of the cresols which were present in the tar before distillation.

#### DEFINITIONS RELATING TO TESTS

*Total Bitumen.*—That portion of bituminous materials, consisting of hydrocarbons and their non-metallic derivatives, which is completely soluble in carbon disulphide.

*Asphaltenes.\* ‡*—The components of the bitumen in petroleum, petroleum products, malthas, asphalt cements, and solid native bitumens which are soluble in carbon disulphide but insoluble in paraffin naphthas. Note: The paraffin naphtha most generally used for determining the percentage of asphaltenes has a gravity of from 86° to 88° Baumé, with at least 85% distilling between 35° and 65° C.

*Carbenes.\* ‡*—The components of the bitumen in petroleum,

petroleum products, malthas, asphalt cements, and solid native bitumens which are soluble in carbon disulphide but insoluble in carbon tetrachloride.

*Naphthalene*.—A solid crystalline highly volatile hydrocarbon occurring principally in tars, and having the chemical formula  $C_{10}H_8$ .

*Free Carbon in Tars*.\*†—Organic matter which is insoluble in carbon disulphide.

*Fixed Carbon*.\* †—The organic matter of the residual coke obtained upon burning hydrocarbon products in a covered vessel in the absence of free oxygen.

*Normal Temperature*.\* †—As applied to laboratory observations of the physical characteristics of bituminous materials, is  $25^{\circ}$  C. ( $77^{\circ}$  F.). Note: In the calibration of the volume of containers and the gauging of their contents,  $15.5^{\circ}$  C. ( $60^{\circ}$  F.) is customarily considered as normal temperature.

*Consistency*.\* †—The degree of solidity or fluidity of bituminous materials.

*Viscosity*.†—The measure of the resistance to flow of a bituminous material, usually stated as the time of flow of a given amount of the material through a given orifice.

*Penetration*.†—The consistency of a bituminous material expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time, and temperature. When the conditions of test are not specifically mentioned, the load, time, and temperature are understood to be 100 grams, 5 seconds,  $25^{\circ}$  C. ( $77^{\circ}$  F.), and the units of penetration to indicate hundredths of a centimeter.

#### DEFINITIONS RELATING TO USE

*Dust Preventive*. Material applied to a road surface for preventing the formation or dispersion under traffic of dust.

*Bituminous Surface*.†—A superficial coat of bituminous material with or without the addition of stone or slag chips, gravel, sand, or material of similar character.

*Carpeting Medium*.—Bituminous material applied to a road surface primarily for protecting the road proper from the wear

and tear of traffic through the formation of a mat or carpet covering.

*Carpet.*‡—A bituminous surface of appreciable thickness, generally formed on top of a road or pavement by the application of one or more coats of bituminous material with gravel, sand, or stone chips added.

*Seal-Coating Material.*—Bituminous material applied to the surface of a bituminous road primarily for the purpose of filling surface voids and producing a smooth, uniform wearing surface.

*Bituminous Cement.*—Bituminous material introduced into the road structure primarily for the purpose of cementing together and holding in place the fragments of mineral aggregate.

*Bituminous Macadam Pavement.*—One having a wearing course of macadam with the interstices filled with a bituminous binder applied by the penetration or pouring method.

*Bituminous Concrete Pavement.*‡—One composed of stone, gravel, sand, shell, or slag, or combinations thereof, and bituminous materials incorporated together by mixing methods. Note: This definition includes sheet-asphalt pavements which are not ordinarily considered as bituminous concrete.

*Sheet-Asphalt Pavement.*‡—One having a wearing course composed of asphalt cement and sand of predetermined grading, with or without the addition of fine material, incorporated together by mixing methods.

*Sheet-Asphalt Topping.*—The bituminous aggregate used in the construction of the wearing course of a sheet-asphalt pavement.

*Binder Course.*—A rather coarse bituminous aggregate containing a relatively small percentage of bitumen, commonly used as an intermediate course between the foundation and wearing course of a sheet-asphalt pavement.

*Bituminous Filler.*—Bituminous material primarily used for filling the joints in brick, block, concrete, or other pavements.

## TYPES OF BITUMINOUS MATERIALS

There are a great many types of bituminous materials, but only a comparatively few are of direct interest to the highway engineer at present. In classifying these types it is most con-



venient to consider the crude materials. Of these there are two main groups, native and artificial or pyrogenous.

The native bituminous materials may be either liquid, semi-solid, or solid. The liquids are known as petroleum and malthas and run from thin fluids to those which are very viscous and almost semisolid. There are two well-defined classes of petroleum—paraffin and asphaltic. There is also an intermediate class commonly termed semi-asphaltic. The semisolid and solid native bituminous materials of direct interest divide themselves into two classes, asphalts and rock asphalts. Most of the native bituminous materials from a given locality possess certain chemical or physical properties which serve to identify them and to distinguish them from materials obtained from other localities. They are therefore most conveniently classified according to the location from which they are obtained. We thus speak of California, Texas, and Mexican petroleum, Trinidad and Bermudez asphalt, Kentucky and Sicilian rock asphalt, etc.

The crude artificial or pyrogenous bituminous materials are fluid products known as tars. Like the petroleum, they run from thin liquids to those which are very viscous. There are two classes of tars of direct interest to the highway engineer, coal tars and water-gas tars. The former, as their name implies, are derived from bituminous coal by a process of destructive distillation. Water-gas tars are, however, derived from petroleum or petroleum products by a cracking process in the manufacture of carburetted water gas. As practically all crude tars are by-products, they may be further classified according to the process by which they are obtained and the type of apparatus involved in the process. We thus have under coal tars two groups: (1) gas-house and (2) coke-oven; and under the first group horizontal retort, inclined retort, and vertical retort tars. This method of classification is particularly convenient as a means of identification, as the process of manufacture and type of apparatus influence the character of the tar much more than does the character of the original material which is destructively distilled or cracked to produce them.

In most cases crude bituminous materials are much easier

to identify than are the products refined or manufactured from them for use in highway work. This is due to the fact that many refined products consist of a blend or combination of two or more types. Certain distinguishing characteristics of the original crude materials may, however, often be discovered in a refined product and thus serve to identify at least its predominating source. A knowledge of the characteristics of crude bituminous materials is therefore of considerable value in the interpretation of the results of tests made upon the refined products directly used in highway engineering.

The following outline, while far from complete, will serve as a general classification of crude bituminous materials of most interest to the highway engineer.

## CLASSIFICATION OF CRUDE BITUMINOUS MATERIALS

### I. NATIVE.

#### *a.* Fluids.

##### 1. Paraffin Petroleums:

Pennsylvania.

Ohio-Indiana or Lima.

##### 2. Semi-asphaltic and Asphaltic Petroleums:

Mid-Continent.

Texas.

Southern Illinois.

California.

Mexican.

Trinidad.

#### *b.* Semisolids and Solids.

##### 1. Asphalts:

Trinidad.

Bermudez.

Cuban.

Gilsonites.

Grahamites.

##### 2. Rock Asphalts:

Kentucky.



Uvalde.  
Sicilian—Ragusa.  
Val de Travers.  
Seyssel.

## II. PYROGENOUS.

### *a.* Produced from Coal.

#### 1. Gas-House Coal Tars:

Horizontal Retort.

Inclined Retort.

Vertical Retort.

#### 2. Coke-Oven Tars:

Koppers.

Semet-Solvay.

United Otto.

Otto-Hoffman.

Rothburg.

### *b.* Produced from Petroleum.

#### 1. Oil-Gas Tars.

#### 2. Water-Gas Tars.

## REFINING PROCESSES

The ordinary refining processes employed in the manufacture of bituminous road and paving materials may be classified as follows:

### I. Removal of Non-bituminous Impurities:

*a.* Sedimentation.

*b.* Dehydration.

### II. Distillation:

*a.* Fractional Distillation.

*b.* Cracking.

*c.* Destructive Distillation.

### III. Oxidation or Blowing.

### IV. Fluxing:

*a.* Fluxing with Residuals.

*b.* Fluxing with Distillates—Cutting-Back.

*c.* Emulsification.



*Sedimentation.*—Water is the most common impurity in both fluid and solid crude bituminous materials. Mineral matter, vegetable matter, and occluded gases are other natural occurring impurities. As most of these impurities are insoluble in the bitumen proper, and as they differ from the bitumen in specific gravity, they may be removed wholly or in part by the process of sedimentation or separation by gravity. In the more fluid bituminous materials, natural sedimentation occurs during storage in large tanks. In the case of crude petroleums, the water and mineral matter settle to the bottom, leaving practically pure bitumen above. In the case of tars which are heavier than water, the water rises to the top, leaving the tar, together with certain impurities, such as free carbon, which will not separate by sedimentation, in the bottom of the tank. Very viscous fluids persistently retain a small amount of water, and the separation of this water may often be expedited by equipping the storage tank with steam coils, by which means the temperature of the bituminous materials may be raised so as to produce a greater degree of fluidity. It frequently happens, however, that even under such conditions all of the water will not separate, and the material is then subjected to a process of dehydration prior to its further refining. When the solid native bitumens contain impurities they invariably have to be heated to a rather high temperature before they become sufficiently fluid to separate from these impurities. In order to prevent local overheating they are usually agitated during the process. Water and gas are thus driven off, some of the mineral matter settles to the bottom, and the larger fragments of vegetable matter rise to the surface and are skimmed off. The mineral matter is, however, sometimes so finely divided that it will not readily settle out, and agitation further hinders this separation. Both finely divided mineral and vegetable matter therefore commonly occur in refined asphalt.

*Dehydration.*—Dehydrating processes are designed primarily for the removal of water in bituminous material which will not completely separate water by sedimentation. It is desirable to do this prior to distillation because of the fact that the presence

of water creates a tendency to foam when the mass of bituminous material is heated to about the temperature of boiling water. Dehydrating plants vary considerably in design, but those more commonly used for petroleum are known as topping plants. In this device the oil is pumped, frequently under pressure, through a length of pipe containing a great many abrupt bends, so that the path of the oil is exceedingly devious. The pipe work is set in a furnace so that it may be suitably heated, and as the oil under pressure passes through this pipe it is heated to a temperature above that of boiling water. This pipe discharges in a spray into a large expansion chamber where the water and more volatile constituents separate in the form of vapor, which is condensed in a coil for the recovery of light condensable hydrocarbons. The hot oil passes through another pipe direct to the still or storage tank. One of the most successful methods for dehydrating viscous tars is to cause the tars to flow in thin films over heated baffle-plates placed in an air-tight chamber to which vacuum is applied. Here the water and more volatile constituents pass off without causing the tar to foam dangerously, and the volatile products are then condensed and recovered by suitable means.

*Fractional Distillation.*—Fractional distillation is that form of distillation in which the original constituents of a mixture are separated mechanically and without chemical change. Petroleum and tars are often subjected to fractional distillation in the manufacture of bituminous materials of interest in highway engineering. The distillation is usually made upon the crude product which has been partially separated from its impurities by sedimentation and dehydration. Two main classes of products are obtained by distillation: 1. Distillates which pass over into the receiver, and, 2. Residues which remain in the still. The former, with a few exceptions, are of comparatively little interest from the standpoint of highway engineering. The residues, however, often constitute the materials directly used in the treatment or construction of highways. As the temperature of distillation increases, more and more of the lighter products are removed and the residues in the still become more



and more viscous until finally upon cooling they may become solid. In producing these residues for highway work, it is of prime importance that the material suffer no chemical change due to high temperatures; in other words, that the hydrocarbons present should be merely separated and not broken up chemically.

*Cracking.*—At high temperatures most hydrocarbons tend to break up into other compounds. This is known as cracking, and, as it often injures the character of the material being distilled, precautions are ordinarily taken to prevent cracking. The most common means of securing a separation of the higher boiling constituents is to force steam through the mass of material in the still so as to mechanically carry over certain compounds at temperatures lower than their normal boiling point. Certain tests are used in connection with the examination of bituminous materials to determine whether or not they have been cracked, and thereby injured by overheating during the refining process.

*Destructive Distillation.*—Destructive distillation is not, properly speaking, a refining process, but it is of particular interest in connection with the incidental manufacture of tars from coal and other materials. It consists of a complete breaking down of the original compounds present in the material distilled by the action of very high temperatures. This results in the formation of certain condensable products which are known as tars. In fact, the formation of tars is dependent upon cracking or destructive distillation of some other material. When these tars, however, are refined for the purpose of producing bituminous road and paving materials, as much care is necessary to prevent cracking during the fractional distillation as in the case of petroleum products. In other words, while the tar itself is produced by destructive distillation, it in turn must not be destructively distilled or cracked when making refined tar products.

*Blowing.*—Certain classes of bituminous materials such as fluid petroleum residuums may be transformed into semisolid or solid products without material loss in volume or weight by the



use of air which is injected into the residuum previously heated to a temperature between 400 and 450° F. The air at this temperature causes certain chemical reactions to take place which tend to unite two or more fluid compounds into single, more viscous, or semisolid compounds. This process is known as the blowing process, and petroleum residuums which are blown are known as blown petroleums. The operation is frequently conducted in open or semi-open kettles, although the same result may be secured by injecting air into the still at a certain stage of the distillation process and then controlling the temperature so that a true distillation will not take place.

*Fluxing.*—The solid native bituminous materials are usually too hard to be used directly in highway engineering. After preliminary refining, which consists of the partial or complete removal of impurities, they are softened to the desired consistency by combining or fluxing them with fluid or relatively soft petroleum residuums. The fluxing process is ordinarily conducted in an open tank or kettle, which may be equipped with steam coils or heated by direct fire. The hard bitumen is first melted in the kettle, after which the heated petroleum residuum or flux is run in, in amount sufficient to produce a finished product of the desired consistency. Rather prolonged agitation of the contents of the kettle is required to secure an absolutely uniform product. Such agitation may be obtained by means of a mechanical stirring device, or with steam or air. Mechanical agitation is preferable in certain respects, but it is not as efficient as the other two methods. Agitation with steam is desirable from many standpoints, but invariably results in the mechanical removal of some of the lighter constituents present. Air agitation, on the other hand, causes some oxidation to take place, although the temperatures employed are not as high as that at which such oxidation becomes very pronounced. In both steam and air agitation, however, some allowance in the proportions of flux to be used should be made for unavoidable hardening. This factor has to be taken into account at a paving plant where the fluxing process is employed.

*Cutting-Back.*—Distillates are sometimes used as fluxes for

the harder bituminous materials, which are then said to be cut-back. The term "cut-back" was originally applied to the practice of first distilling a bituminous material until the residuum was hard and brittle, in order to obtain certain valuable products from the heavier distillates, after which a portion of the distillate was run back or incorporated with the hard residuum to bring it to the desired consistency. It has, however, gradually come to be used to denote the use of a distillate as a flux for any natural or artificial residuum.

*Emulsification.*—All bitumens are practically insoluble in water. If, however, a strong solution of soap is used, the soap acts as a medium for holding the bitumen and water together in suspension or emulsion, and, if properly made, such emulsion may be further mixed or diluted with water without causing separation. Thus emulsions of petroleums, asphalts, and tars may be obtained under suitable conditions, and such emulsions are used to some extent in the surface treatment, and even in the construction of highways.

## CLASSIFICATION OF BITUMINOUS ROAD AND PAVING MATERIALS

*Classification According to Use.*—Bituminous materials as directly used in highway engineering may be conveniently classified according to use as follows:

- I. Surface Treatment:
  - a. Dust Preventives.
  - b. Carpeting Mediums.
  - c. Seal-Coating Materials.
- II. Incorporation in the Pavement Structure:
  - a. Bituminous Cements.
  - b. Bituminous Fillers.
  - c. Bituminous Aggregates.
  - d. Impregnating Materials for Wood Block.

Materials used in both surface treatment and the pavement structure may be either of native or pyrogenous origin. This is one of the first things to ascertain from a laboratory sample.



The use of further tests will then in many cases be determined by the specific purpose for which the material is to be used or is supposed to be suitable.

*Dust Preventives.*—Dust preventives are usually relatively thin fluids which may be applied without preheating. They should be susceptible to a light uniform distribution so as to saturate the dust particles on the road, but not penetrate the road surface to any extent. They need not necessarily possess cementitious properties nor develop cementitiousness after application, as they are not primarily intended for the construction of bituminous carpets, and if properly used the treated road rarely requires a covering of fine mineral matter. To be of maximum service, they should not volatilize rapidly to any extent under ordinary atmospheric conditions. Dust preventives in most common use are:

1. Crude and Topped Petroleums.
2. Heavy Petroleum Distillates.
3. Petroleum Emulsions and Emulsifying Oils.
4. Crude and Dehydrated Tars.

*Carpeting Mediums.*—Carpeting mediums are usually viscous fluids and often have to be heated before they can be satisfactorily applied to the road surface. They should be of such normal consistency that when applied they will not at once become so viscous as to prevent their uniform distribution and adherence to a properly prepared surface of the type of road upon which they are used. They should possess or develop shortly after application sufficient cementitiousness to bind together and hold in place a covering of sand, fine gravel, or stone chips, with which they combine to form the mat or carpet. They should furthermore waterproof the road surface. If they contain a base of high binding value they may well carry a relatively high percentage of volatile constituents, which after application rapidly evaporate and leave practically a bituminous cement in place. The carpeting mediums in most common use are:

1. Residual Petroleums.
2. Cut-back Asphalt Cements.
3. Residual or Refined Tars.



*Seal-Coating Materials.*—Seal-coating materials are in reality a superior type of carpeting medium applied to bituminous-constructed roads. In many cases they are the same type and grade of bituminous cement used in the construction of the underlying wearing course. While they will adhere to a clean bituminous surface, they cannot be satisfactorily used in the surface treatment of ordinary gravel or macadam roads.

*Bituminous Cements.*—Bituminous cements are very viscous or semisolid materials whose function is to bind and waterproof at least the wearing course of the road structure. Their requisite binding strength and degree of hardness are largely dependent upon the size and grading of the mineral aggregate which they cement together. In general, the finer the aggregate the greater becomes the necessary degree of solidity and binding power of the bituminous cement. For the finer aggregates, therefore, high susceptibility to temperature changes is to be particularly avoided. Thus refined tars, which are the most susceptible to temperature changes of all bituminous materials, may be satisfactorily used in the construction of bituminous macadam or coarse aggregate bituminous concrete, but are not well suited for use in the fine aggregate concretes. Bituminous cements in most common use at present are:

1. Oil Asphalts.
2. Blown Petroleums.
3. Fluxed Native Asphalts.
4. Very Viscous Refined Tars.

*Bituminous Fillers.*—Bituminous fillers may be of the nature of relatively hard bituminous cements or mixtures of bituminous materials with very finely divided mineral matter. In the former case they are most frequently heated to a fluid condition and then poured into the joints of the pavement. In the latter case they are called prepared fillers and are often manufactured in the form of strips of proper width and thickness to place in the joints. The surface is then sometimes sealed with a hot iron. Bituminous fillers should adhere to the sides of the joints and waterproof them. In cold weather they should not become so hard as to chip under traffic, and in hot weather, when the pave-

ment expands, they should not become so fluid as to bleed and produce a sticky surface. The principal types of bituminous fillers are:

1. Blown Petroleums and Asphalt Cements.
2. Tar Pitches.
3. Mixture of Asphalt Cements with Finely Divided Silica, Limestone Dust, or Clay.

*Bituminous Aggregates.*—Bituminous aggregates are ordinarily used to produce directly the wearing course of a pavement. Many of them are known by names which have come to indicate certain limitations of grading of the mineral aggregate; others are known according to their origin or method of manufacture. Patents have been granted upon various combinations of mineral matter with bituminous cements, and some of these combinations are used under patented or trade names. The important types of bituminous aggregates are:

1. Binder Course.
2. Sheet Asphalt Topping or Surface Mix.
3. One Size Broken Stone Concrete.
4. Coarse Graded Concrete.
5. Fine Graded Concrete.
6. Bituminous Gravel Concrete.
7. Bituminous Earth.
8. Rock Asphalt.
9. Asphalt Block.

*Impregnating Materials.*—Impregnating materials are ordinarily used in the treatment of wood block. They should be sufficiently fluid and free from suspended matter to impregnate the block thoroughly and should possess sufficient antiseptic and germicidal properties to prevent decay of the wood. They should also waterproof it to a very considerable extent. When used in proper amount they should not bleed from the wood in warm weather and produce a sticky surface. To be of lasting value, they should contain a relatively low percentage of highly volatile constituents. Tars and tar products are ordinarily used for this purpose. Recently asphalt cements have been used to impregnate building or paving brick, but such products are not

usually termed impregnating materials. The tar products of most importance are:

1. Tar Distillates.
2. Fluid Refined Tars.
3. Mixtures of Refined Tars with Tar Distillates.

## LABORATORY SAMPLING, PREPARATION, AND MANIPULATION

*Representative Samples.*—Samples of bituminous materials as received by the laboratory are presumably representative of the entire bulk of material sampled, whether it be the contents of a storage tank, a single batch of manufactured product, or a consignment of material shipped in tank cars, barrels, or drums. Too often this is not the case, but methods of plant and field sampling do not come within the scope of this manual, and it must therefore be assumed that the original samples have been properly taken. If evidence to the contrary exists, as indicated by the presence of dirt, chips of wood, or other apparently extraneous materials, the laboratory is usually justified in discarding such samples, and insisting upon fresh ones, as it is exceedingly difficult, and often impossible, to remove the extraneous material prior to making a laboratory examination without in some way changing the characteristics of the original material. Rejection may also properly be made of samples which have become contaminated or altered through careless or wrong methods of packing and shipping. Thus it is not at all unusual to find that a sample has been in direct contact with excelsior, sawdust, or wrapping paper which has adhered to or mixed with it during shipment. The presence of such extraneous matter may vitiate many of the tests which it may be found necessary or desirable to make upon the material.

*Size of Samples.*—While a complete set of tests may often be made upon a much smaller sample, it is usually desirable that the laboratory have at hand not less than a one-quart sample of petroleum, asphalt, and tar products; not less than two pounds of rock asphalt or fine bituminous aggregate, and



about five pounds of coarse bituminous aggregate. In certain cases two or more samples may be submitted from a single shipment or lot of material with the understanding that a few tests will be made upon each to determine whether the individual samples are of uniform character, and that a complete set of tests will then be made upon a composite sample prepared in the laboratory from equal proportions of the individual samples submitted. In such cases the size of the individual samples should be such as to allow for the tests of uniformity, usually specific gravity and consistency, with a surplus amply sufficient to afterward prepare a single composite sample of the same size as though only one sample were submitted. For daily routine laboratory checks of plant or field practice, the most desirable size of sample will depend upon just what check tests are required. While the laboratory may not always need enough material to make duplicate tests, it should always have at hand a sufficient amount for that purpose, and in addition enough to file away for possible future reference.

*Containers.*—For fluid products, a one-quart rectangular can with small, tight-fitting screw top is preferable. For very viscous and soft semisolid products, a one-quart cylindrical can with tight-fitting pry lid, such as commonly used for paints, will be found convenient. For the harder semisolid and solid products, a shallow rectangular or cylindrical can with ordinary box lid top is suitable.

*Identification.*—The laboratory should insist that all samples submitted bear on the outside of the container marks to sufficiently identify them. This would ordinarily include the name and address of the shipper, the tank or car number from which the sample was taken, the location and type of work in which it is to be used, and in certain cases the name of the manufacturer. If it is to be examined for conformity with any particular specification, this fact should also be indicated. Upon receipt of the sample this information should be immediately recorded in the laboratory note-book for the purpose of identifying the sample with the results of tests. An example of such identification is shown under method of keeping laboratory records.

*Laboratory Sampling.*—Even when truly representative samples of bituminous materials are submitted to the laboratory representative test results may not be obtained by skilled operators unless special precautions are taken to insure that that portion of the sample used in a given test is representative of the entire sample. Thus segregation of certain constituents may take place during shipment or upon standing, and when this is likely to occur the first rule to be observed is to thoroughly mix the contents of the sample can before removing a portion for test. In the case of viscous liquids, semisolids, and solids, this will usually necessitate heating, in which case extreme care should be taken that no loss by overheating takes place. The material should be mixed at the lowest practicable temperature, and preferably in the original container. In the case of bituminous aggregates the mass of material should be slowly warmed in a shallow pan until the bituminous cement has sufficiently softened to allow its disintegration without fracture of the mineral constituents. When composite samples are to be prepared, equal quantities of the individual samples should be weighed out in a new container and thoroughly mixed at the lowest practicable temperature. Mixing may ordinarily be accomplished with a stirring rod, kitchen knife, or kitchen spoon, but in the case of crude products, when it is desired to recombine the bituminous material with water which has separated, recourse may be had to more violent agitation and a small egg-beater will often be found useful for this purpose.

*Equipment for General Manipulation.*—For general laboratory manipulation such as heating, preparing, and transferring bituminous materials, the more expensive equipment of the chemical laboratory, such as beakers, flasks, porcelain dishes, spatulas, etc., may be replaced by cheaper kitchen utensils and hardware. It will be found convenient to have at hand a number of metal saucepans, stamped metal cups free from soldered joints, kitchen knives, and large heavy metal spoons. The use of agate or enameled ware for heating bituminous materials should in general be avoided owing to its tendency to chip and flake with use, thus contaminating the material which is being manipulated.



An assortment of stamped tin boxes of various sizes and shapes, a number of heavy glass stirring rods, and a pair of nickel crucible tongs are also useful for general laboratory handling. A Bunsen burner with tripod and asbestos board square and a gas or electric hot plate are ordinarily sufficient for heating purposes, although a steam bath and a hot-air oven which may be maintained at a low temperature may also often be used to advantage in preparing samples for analysis.

## LABORATORY REPORTS AND RECORDS

*Laboratory Notes.*—For recording observations, noting analytical data, and making calculations, the laboratory operator will usually find some form of note-book more convenient than the card system. Loose-leaf books holding letter-size sheets are in many respects most convenient, but when any necessity may exist for making a chronological record, as, for instance, in connection with court cases, a bound book is the only style which should be used, and the record should be made and dated in ink. Too frequently the laboratory note-book constitutes at best but a slovenly record, and in many cases is decipherable to the operator alone, and then only while the work is fresh in his mind. Little excuse exists for such practice, as it does not require much more time to keep a neat than a careless note-book. Where a large amount of routine work is being conducted, rubber stamps for individual tests, leaving blanks for numerical results, will be found useful.

*General Information.*—Immediately upon receipt of a sample by the laboratory it should be given a serial number, and a dated record should be made of the identification which it bears. Sufficient space should then be left below this descriptive matter to accommodate all of the analytical data which it is expected will be required. If later it is found that sufficient space has not been allowed for this purpose, a note should be inserted that the remaining data will be found upon a subsequent page, the number of which is given. Before continuing the notes on this page the serial number of the sample should



be shown at the top. A suggested form of recording general information is as follows:

No..... Material.....Date.....  
 Submitted by.....  
 Identification marks.....  
 To be examined for.....  
 .....  
 .....

Following the general information it may often be found desirable to insert a brief description of the material, together with a statement of the condition in which it is received.

*Analytical Data.*—Analytical data should be shown in the note-book in sufficient detail to indicate the various operations involved. All calculations should be shown in the notes and not made upon a separate slip of paper or scratch pad. Errors should not be erased but should be crossed out. An example of a brief but complete record of an analytical operation is as follows:

## TOTAL BITUMEN

Wt. Flask + 12.5044	Wt. Crucible + 20.8416
Wt. Flask 10.9032	Wt. Crucible 20.6863
Wt. Sample 1.6012	
	Wt. Insol. Residue 0.1553
1.6012) 0.1453 (0.0907 = 9.07% org.	
0.144108	Wt. Ignited Cruc. 20.6963
1192	Wt. Crucible 20.6863
1.6012) 0.0100000 (0.0062 = 0.62% Ash	Wt. Ash 0.0100
96072	
39280	Wt. Organic Insol. 0.1453
9.69	
90.31 bit.	
100.00	
Total bitumen.....	90.31%
Organic matter insoluble.....	9.07%
Ash.....	0.62%
Total.....	100.00%

*Laboratory Reports.*—The form and substance of a laboratory report should be largely governed by circumstances.

For strictly routine work of any description a printed form may usually be devised which will reduce to a minimum the data which must be added to make the report complete. Where the work is not of a routine nature the matter of form may not be so readily handled. In either case, however, certain information should be included which may be covered by a set form. As an example may be taken the general form adopted by the United States Office of Public Roads and Rural Engineering:

UNITED STATES DEPARTMENT OF AGRICULTURE

Office of Public Roads and Rural Engineering

Washington, D. C.

Sample No.....Date.....  
 Report on.....  
 Identification Marks.....  
 Known as.....  
 Submitted by.....  
 .....  
 Examined for.....  
 .....  
 .....  
 .....  
 Remarks.....  
 .....

After "Report on" the type of material should be inserted, such as Oil Asphalt, Refined Tar, Bituminous Aggregate, etc. After "Known as" is given the trade name, if the material has one, or any other identifying name. After "Examined for" the purpose of the examination is stated, such as identification, suitability for a given purpose, conformity with specifications, etc. Then should follow the analytical data in tabular form, and after "Remarks" should be stated any facts of peculiar interest, particularly with reference to the purpose for which the material is examined. In routine reports where it may be desirable to draw particular attention to certain characteristics, a



rubber stamp with arrow or pointing finger-sign may be used with red ink immediately before or after the analytical result to which attention is called. An explanation of the meaning of this sign should then be given under remarks. Thus when a material is examined for conformity with a given specification, the stamp may be used to call attention to all analytical results which do not fall within the specification limits. Typical examples of routine reports are as follows. In certain cases it may be found more desirable to submit a report on a card form than on paper. If paper be used it is preferable to have the form of ordinary letter size for convenient filing by the recipient. Unusual sizes should be avoided.

## JONES AND BROWN

CHEMICAL ENGINEERS

563 Broadway

New York, July 1, 1916

Sample No. 9436

Report

on

Shipment of Asphalt Cement

Submitted by Wm. A. Smith, City Engineer, Greenville, N. J.

Identification. Three samples marked A, B, and C respectively. "Cemento Asphalt" C. R. R. of N. J. 36074.

Examined for conformity with Greenville specifications, Sheet Asphalt Pavement, Mar. 1, 1916.

Sample	A	B	C
Specific Gravity 25°/25° C.	1.045		1.043
Penetration 25° C., 100 g., 5 sec.	58	56	57

## Composite Sample

Specific Gravity 25°/25° C., average (2) . . . . .	1.044
Flash Point (open cup) . . . . .	245° C +
Melting Point (ring and ball) . . . . .	50° C.
Penetration 25° C., 100 g., 5 sec., average (3) . . . . .	57
Penetration 0° C., 200 g., 1 min. . . . .	18
Loss 163° C., 5 hrs., 50 g. . . . .	0.7%
Penetration residue 25° C., 100 g., 5 sec. . . . .	34
Total bitumen (soluble in CS <sub>2</sub> ) . . . . .	99.90%
Organic matter insoluble . . . . .	0.05%
Inorganic matter insoluble . . . . .	0.05%
	<hr/>
	100.00%

Bitumen insoluble in 86° B. naphtha . . . . .	28.5%
Bitumen insoluble in carbon tetrachloride . . . . .	0.0%
Fixed carbon . . . . .	18.5%

Remarks:—This material conforms with specifications.

Respectfully submitted,

JONES AND BROWN.



## JONES AND BROWN

CHEMICAL ENGINEERS

563 Broadway

Sample No. 9872

New York, Aug. 3, 1916

Daily Report  
on  
Sheet Asphalt Topping

Submitted by Wm. A. Smith, City Engineer, Greenville, N. J.

Identification. Sample 16, laid Aug. 2-16, Poplar St. at intersection of  
Third Street. From load received at 11:00 A.M.

Examined for conformity with Greenville Specifications Sheet Asphalt Pavement, Mar. 1, 1916.

	Original	Sand Basis	
Total bitumen (soluble in CS <sub>2</sub> ).....	10.8		
Passing 200 mesh.....	4.2←		
" 100 mesh, retained on 200 mesh...	15.5	18.2	27.3
" 80 " " " 100 " ...	7.7	9.1	
" 50 " " " 80 " ...	25.8	30.3	47.5
" 40 " " " 50 " ...	14.6	17.2	
" 30 " " " 40 " ...	11.0	13.0	25.2
" 20 " " " 30 " ...	6.1	7.2	
" 10 " " " 20 " ...	4.3	5.0	
Total.....	100.00%	100.0%	100.0%

Remarks:—This sample shows a decided deficiency in filler.

Note:—↑ Indicates variation from specification.

Respectfully submitted,

JONES AND BROWN.

*Laboratory File Records.*—It is highly desirable that the laboratory retain a carbon copy of all reports which it makes. Under ordinary conditions these copies may be conveniently kept in a vertical letter file in tabbed folders bearing the name of the locality from which the material is submitted, or the name of the party submitting the sample. In addition it is highly advantageous to keep a card-abstract file according to the laboratory serial number and to cross-reference this file according to type of material and the name of the manufacturer or the party who submits samples. The most convenient and efficient system for any particular laboratory will of course

depend upon the general character of the work that the laboratory is required to do.

In the case of the card-abstract serial-number file, a 5" by 8" card is suggested, bearing the following information in addition to the abbreviated analytical results.

Serial Number.....

Material.....

Submitted by.....

Identification.....

Examined for.....

Date received.....Date reported.....Analyst.....

.....





## PART II—METHODS OF TESTING

### DENSITY TESTS

#### SPECIFIC GRAVITY

##### *Basis of Determination*

The specific gravity of most bituminous materials used in highway engineering is based upon the relative weights of equal volumes of the bituminous material and water at normal temperature, and is usually expressed as "Specific Gravity  $25^{\circ}/25^{\circ}\text{C.}$ " As the weight of a given volume of any material varies at different temperatures, the temperature basis of comparison should always be indicated for very accurate work. A few bituminous materials, such as certain tar distillates, separate solid matter, mostly naphthalene, at  $25^{\circ}\text{C.}$ , and as this makes it difficult to determine the specific gravity at that temperature, the determination is sometimes made at a temperature sufficiently high to insure complete fluidity. When this is done, water at the same temperature or at  $25^{\circ}\text{C.}$  may be taken as unity. In either case the basis of comparison should be indicated. Thus if the weight of a given volume of the material is obtained at  $X^{\circ}\text{C.}$  and compared with water at  $25^{\circ}\text{C.}$ , the basis of comparison should be expressed as follows: "Sp. Gr.  $X^{\circ}/25^{\circ}\text{C.}$ " An older temperature basis of comparison, which is still used to a considerable extent by manufacturers and also by the United States Government, in the calibration of the volume of tank cars and other containers, is  $15.5^{\circ}\text{C.}$  ( $60^{\circ}\text{F.}$ ). If the coefficient of expansion of the material is known and the specific gravity basis of comparison is given, it is very easy to translate any given determination to terms of another basis of comparison. Such a method is commonly employed by manufacturers who for the

sake of simplicity and rapidity prefer to make use of a hydrometer in the case of many materials which have to be first rendered more fluid by the application of heat. In reporting the specific gravity of fluid bituminous materials it is not uncommon to express results to four places to the right of the decimal point. Three decimal places is usually reported in the case of semisolid and solid materials, owing to the fact that the limit of accuracy is exceeded beyond this point.

### *Hydrometer Method*

#### *Equipment:*

1 hydrometer jar approximately 35 centimeters long and 5 centimeters in diameter. (Fig. 1a.)

1 1-pint tin cup, seamless type. (Fig. 1b.)

1 enamelware dish approximately 2 inches deep and 9 inches in diameter. (Fig. 1c.)

1 chemical thermometer reading from  $-10^{\circ}$  C. to  $110^{\circ}$  C. (Fig. 1d.)

1 set of hydrometers—those with a double scale at  $15.5^{\circ}$  C. ( $60^{\circ}$  F.)—one for Baumé and one for a direct specific gravity reading to the third decimal place—are convenient. (Fig. 1e.)

1 hydrometer reading from 0.800 to 0.900 specific gravity.

1 hydrometer reading from 0.900 to 1.000 specific gravity.

1 hydrometer reading from 1.000 to 1.200 specific gravity.

1 hydrometer reading from 1.200 to 1.400 specific gravity.

*Method.*—The specific gravity of thin fluid bituminous road materials is most readily determined by means of a hydrometer when a sufficient quantity is available for the purpose. The Baumé scale for liquids lighter than water is commonly used by petroleum refiners, and many oil products are sold upon a Baumé degree basis. The Baumé scale for bituminous materials heavier than water is, however, seldom used.

This test is made with the above-mentioned apparatus by first pouring a sufficient quantity of the material into the tin cup, which is then placed in the large dish containing cold

or warm water as occasion may require. The material in the cup should be stirred with the thermometer until it is brought to a temperature of  $25^{\circ}\text{C}.$ , after which it should be immediately poured into the hydrometer jar and its gravity determined by means of the proper hydrometer. In case the hydrometer sinks slowly, owing to the viscosity of the material, it should be given sufficient time to come to a definite resting point, and this point should be checked by raising the hydrometer and allowing it to sink a second time. The hydrometer should never be pushed below the point at which it naturally comes

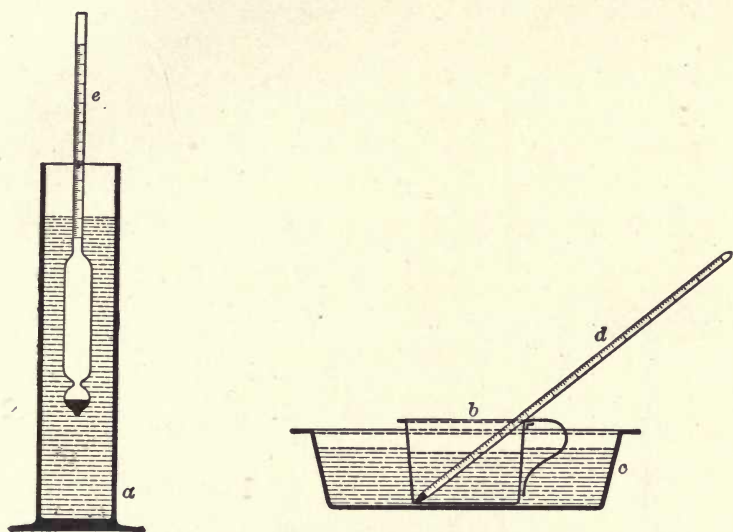


FIG. 1. Hydrometer Method of Determining Specific Gravity

to rest until the last reading has been made. It may then be pushed below the reading for a distance of three or four of the small divisions on the scale, whereupon it should immediately begin to rise. If it fails to do so, the material is too viscous for the hydrometer method, and the pycnometer method should be employed.

Most hydrometers are based upon specific gravity at  $15.5^{\circ}/15.5^{\circ}\text{C}.$  If the material tested is at  $25^{\circ}\text{C}.$ , it is evident that without taking into account the coefficient of expansion of the hydrometer itself, the actual basis of comparison is then



25°/15.5° C. For all practical purposes this may be converted to the 25°/25° C. basis by multiplying the reading obtained by the factor 1.002, which corrects for the decrease in weight of a unit volume of water raised from 15.5° to 25° C. If the specific gravity is obtained at a considerably elevated temperature, the coefficient of expansion of the hydrometer itself should be taken into account as well as that of the water and the material examined.

When double-scale hydrometers are not available, the results obtained by one scale may be transposed to the other by means of the following formulas for liquids lighter than water:

$$\text{Sp. Gr.} = \frac{140}{130 + ^\circ\text{B.}} \text{ at } 15.5^\circ \text{ C.}$$

$$^\circ\text{B.} = \frac{140}{\text{Sp. Gr.}} - 130 \text{ at } 15.5^\circ \text{ C.}$$

From these formulas it will be seen that 1.000 specific gravity is the equivalent of 10° B., and that as specific gravities decrease the degrees Baumé increase.

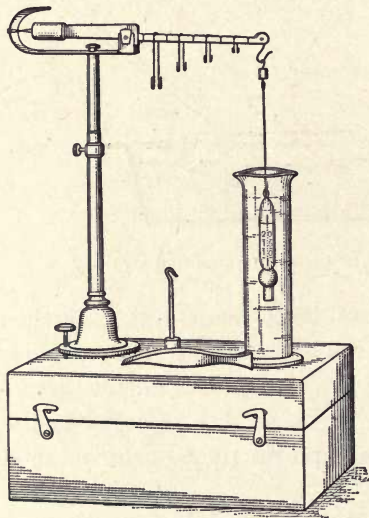


FIG. 2. Westphal Balance

### *Westphal Balance Method*

A Westphal balance (see Fig. 2) is convenient for quickly determining the specific gravity of comparatively small quantities of fluid bituminous materials, particularly fluid distillates. By using a vertical cylindrical container of about 5/8-inch diameter, it may be possible to use this instrument with as little as 15 c.c. of material.

The determination is made by balancing a plummet in the liquid, the scale of the balance and the weights used being so designed as to give direct specific gravity readings. The Westphal balance is usually calibrated for 15.5°/15.5° C. determinations, and the results

obtained are subject to the same corrections as noted under the hydrometer method.

*Sprengel or Nicol Tube Method**Equipment:*

- 1 Sprengel or Nicol tube with fine wire support.
- 1 20-cubic centimeter low-form glass beaker.
- 1 400-cubic centimeter low-form glass beaker.
- 1 chemical thermometer reading from  $-10^{\circ}$  C. to  $110^{\circ}$  C.
- 1 Bunsen burner and rubber tubing.
- 1 iron tripod with wire gauge.
- 1 glass stirring rod.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligrams.
- 1 piece of blotting paper.

*Method.*—This method is well adapted for determining the specific gravity of very small quantities of thin fluid bitumens or such materials as tar distillates containing precipitated solids at normal temperature. In the latter case the determination should be made at an elevated temperature such as  $38^{\circ}$  C. ( $100^{\circ}$  F.) in order to have the material completely fluid.

The Nicol tube, as shown in Fig. 3, may be made with a capacity as low as 0.5 c.c. It may be conveniently suspended by means of a fine wire from the hook on one of the pan supports of the analytical balance. One arm of the tube is drawn out to a fine capillary opening. The other arm is somewhat larger but open, and carries a mark to which the instrument is filled by sucking in the material through the smaller arm. Any excess beyond the mark may be removed by means of a small piece of blotting paper.

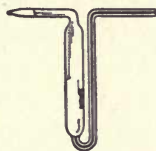


FIG. 3. Nicol Tube

The test is made by first weighing the clean dry tube and then weighing it filled with water at normal or other temperature, according to the desired basis of comparison. The tube is next emptied and thoroughly dried by means of a current of air. It is then filled with the material under examination and again weighed. Small quantities of the material may be conveniently handled in the 20-c.c. beaker, and the larger beaker

filled with water at the desired temperature may be used as a control bath when filling the instrument.

Calling the weight of the empty tube  $a$ , its weight filled with water  $b$ , and its weight filled with the material under examination  $c$ , the specific gravity may be calculated from the following formula:

$$\text{Specific Gravity} = \frac{c - a}{b - a}.$$

### *Pycnometer Method*

#### *Equipment:*

- 1 large metal kitchen spoon.
- 1 steel spatula or kitchen knife.
- 1 Bunsen burner with rubber tubing.
- 1 250-cubic centimeter low-form glass beaker.
- 1 chemical thermometer reading from  $-10^{\circ}\text{C.}$  to  $110^{\circ}\text{C.}$
- 1 special pycnometer. (Fig. 4.)
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

*Method.*—The inconvenience and difficulty of employing the ordinary narrow-neck pycnometer when determining the specific gravity of viscous fluid and semisolid bitumens have led to the use of the special form shown in Fig. 4.



FIG. 4. Hubbard Pycnometer

This pycnometer consists of a fairly heavy, straight-walled glass tube, 70 millimeters long and 22 millimeters in diameter, carefully ground to receive an accurately fitting solid glass stopper with a hole of 1.6 millimeters bore in place of the usual capillary opening. The lower part of this stopper is made concave in order to allow all air-bubbles to escape through the bore. The depth of the cup-shaped depression is 4.8 millimeters at the center. The stoppered tube has a capacity of about 24 cubic centimeters, and when empty weighs about 28 grams. Its principal advantages are (1) that any desired amount of bitumen may be poured in without touching the sides above



the level desired; (2) it is easily cleaned; (3) on account of the 1.6-millimeter bore, the stopper can be more easily inserted when the tube is filled with a very viscous oil than if it contained a capillary opening.

When working with semisolid bitumens which are too soft to be broken and handled in fragments, the following method of determining their specific gravity is employed. The clean dry pycnometer is first weighed empty, and this weight is called *a*. It is then filled in the usual manner with freshly distilled water at 25° C., and the weight is again taken and called *b*. A small amount of the bitumen should be placed in the spoon and brought to a fluid condition by the gentle application of heat, with care that no loss by evaporation occurs. When sufficiently fluid, enough is poured into the dry pycnometer, which may also be warmed, to fill it about half full, without allowing the material to touch the sides of the tube above the desired level. The tube and contents are then allowed to cool to room temperature, after which the tube is carefully weighed with the stopper. This weight is called *c*. Distilled water, at 25° C., is then poured in until the pycnometer is full. After this the stopper is inserted, and the whole cooled to 25° C. by a 30-minute immersion in a beaker of distilled water maintained at this temperature. All surplus moisture is then removed with a soft cloth, and the pycnometer and contents are weighed. This weight is called *d*. From the weights obtained the specific gravity of the bitumen may be readily calculated by the following formula:

$$\text{Specific Gravity } 25^{\circ} \text{ C./}25^{\circ} \text{ C.} = \frac{c - a}{(b - a) - (d - c)}.$$

Both *a* and *b* are constants and need be determined but once. It is, therefore, necessary to make but two weighings for each determination after the first. Results obtained according to the method given above are accurate to within 2 units in the third decimal place, while the open-tube method is accurate to the second decimal place only.

The specific gravity of fluid bitumens may be determined in the ordinary manner with this pycnometer by completely filling

it with the material and dividing the weight of the bitumen thus obtained by that of the same volume of water.

The pycnometer may be readily cleaned by placing it in a hot-air bath until the bitumen is sufficiently fluid to pour. As much is drained out as possible and the interior swabbed with a piece of cotton waste. It is then rinsed clean with a little carbon disulphide, and after drying is again ready for use.

### *Displacement Method*

#### *Equipment:*

- 1 chemical thermometer reading from  $-10^{\circ}$  C. to  $110^{\circ}$  C.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.
- 1 wood or metal platform.
- 1 150-cubic centimeter low-form glass beaker.
- 1 piece of fine silk thread.

*Method.*—For materials which are hard enough to be broken and handled in fragments at room temperature, the following method will prove convenient. A small fragment of the bitumen (about 1 c.c.) is suspended by means of a silk thread from the hook on one of the pan supports, about  $1\frac{1}{2}$  inches above the pan, and weighed. This weight is called  $a$ . It is then

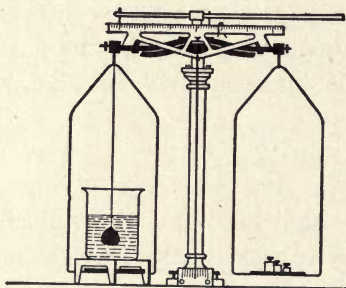


FIG. 5. Displacement Method of Determining Specific Gravity

weighed immersed in water at  $25^{\circ}$  C., as shown in Fig. 5, and this weight is called  $b$ . The specific gravity may then be calculated by means of the following formula:

$$\text{Specific Gravity} = \frac{a}{a - b}$$

This method may be used in the determination of the specific gravity of compressed bituminous aggregates such as sections of bituminous pavement, asphalt block, etc. In the case of coarse aggregates, when it is desirable to run the test upon a large sample, a rough

balance, sufficiently sensitive to accurately give specific gravity determinations to within 2 units in the third significant figure, may be rigged up on the edge of a table, so that the sample may be suspended in a bucket of water from one of the pan supports. As bituminous aggregates may frequently absorb water during the test, it is advisable to determine their apparent specific gravity. This may be done by first weighing the specimen in air and then immersing it in water for 24 hours. The specimen is then removed from the water, rapidly surface-dried with a piece of cloth, and again weighed in air. It is then weighed immersed in water. Calling the original weight in air  $a$ , the weight in air after absorption  $b$ , and the weight in water  $c$ , the apparent specific gravity is calculated by means of the following formula:

$$\text{Apparent Specific Gravity} = \frac{a}{b - c}.$$

### *Method for Coarse Mineral Aggregates*

A convenient apparatus for determining the specific gravity of coarse mineral aggregates is shown in Fig. 6. It consists of a brass cup, "A,"  $4\frac{1}{2}$  inches in diameter and  $6\frac{3}{4}$  inches high, which is fitted with a conical cap, "B." The sides of the cup are turned down at the top one-half the thickness of the metal, forming a shoulder about one inch deep. A similar shoulder cut in the cap provides an absolutely constant volume when the cap is in position. The joint is made water-tight by snapping an ordinary rubber band about  $\frac{1}{2}$  inch wide around it. A glass tube, "C," with a single graduation fixes the volume of the cup. A special form of burette, "D," carrying two bulbs of 200 and 600 c.c. capacity, and a graduated tube of 200 c.c. capacity, capable of being read to the nearest cubic centimeter, is conveniently used for measuring the water, although the water may be weighed from a 1000-c.c. glass flask if desired.

When making a determination, a sample of the material weighing about 1000 grams is first dried to constant weight in an oven at  $110^{\circ}\text{C}.$ , cooled, and accurately weighed to the nearest 0.5 gram. It is then immersed in water for 24 hours, after which it is surface-dried with a towel, reweighed, and immediately introduced into the brass cup. The cap is then placed in position and the cup filled with water from the burette up to the graduation, "C." Knowing

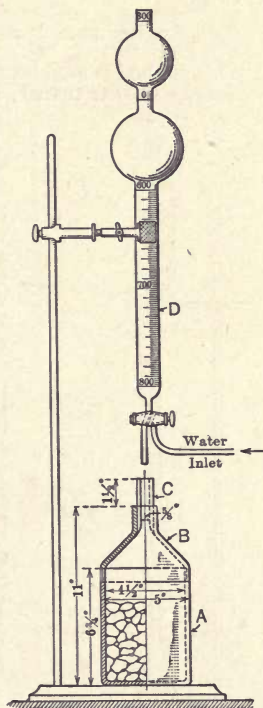


FIG. 6. Hubbard-Jackson Specific-Gravity Apparatus



the volume of the cup, the apparent specific gravity of the material may be calculated as follows: Calling the weight of the dry sample in air  $a$ , the total volume of the cup  $b$ , and the volume of water necessary to fill it after introducing the sample  $c$ , then

$$\text{Apparent Specific Gravity} = \frac{a}{b-c}.$$

When it is desired to obtain as nearly as possible the apparent specific gravity of aggregates consisting of a mixture of coarse and fine particles, it is advisable to separate a weighed sample of the material by means of a  $\frac{1}{2}$ -inch screen and to make an apparent specific gravity determination upon about 1000 grams of the coarse fraction and a true specific gravity determination upon not less than 50 grams of the finer fraction by the method for fine mineral aggregates. The specific gravity of the whole may then be calculated from the following formula, where  $W$  equals the weight per cent of coarse aggregate,  $W^1$  the weight per cent of fine aggregate, and  $G$  and  $G^1$  their respective specific gravities:

$$\text{Approximate Apparent Specific Gravity} = \frac{100}{W/G + W^1/G^1}.$$

This method is used because it is impracticable to surface-dry a large mass of particles smaller than  $\frac{1}{2}$  inch in diameter.

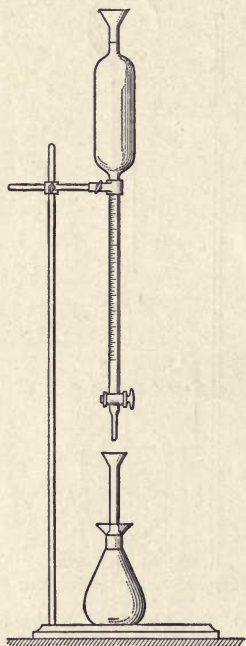


FIG. 7. Jackson Specific-Gravity Apparatus

### *Method for Fine Mineral Aggregates*

The apparatus used in this method is shown in Fig. 7, and consists of an Erlenmeyer flask, fitted with a special form of hollow ground-glass stopper, and having a capacity of 200 c.c. up to the graduation on the stopper tube. A special form of burette with graduations to 0.01 in specific gravity, provided with a bulb of 180 c.c. capacity, is conveniently used for measuring the water displaced. In making the specific gravity determination, the burette is first filled with water to the mark above the bulb. Water is then run into the flask until the burette bulb is about half empty. A sample of sand or other fine aggregate dried to constant weight, and weighing to within 0.01 gram of 50 grams, is then slowly introduced into the flask, after which the hollow ground-glass stopper is inserted and the flask filled with water up to the graduation on the stopper. The specific gravity may then be read directly on the burette. It is important that throughout this test the water be kept at a constant temperature, and that all entangled air-bubbles in the aggregate and on the sides of the flask be removed by agitation.

### *Value of Specific-Gravity Determination*

A specific-gravity determination is commonly made upon all types and grades of bituminous materials, with the exception of loose or uncompressed bituminous aggregates. It is one of the most valuable means of identifying a bituminous material,

particularly in connection with a test of consistency. When considered in connection with other tests it is also often of service in determining the suitability of a material for a given use. In specifications it is used for both purposes and also for the sake of controlling uniformity of supply from a given

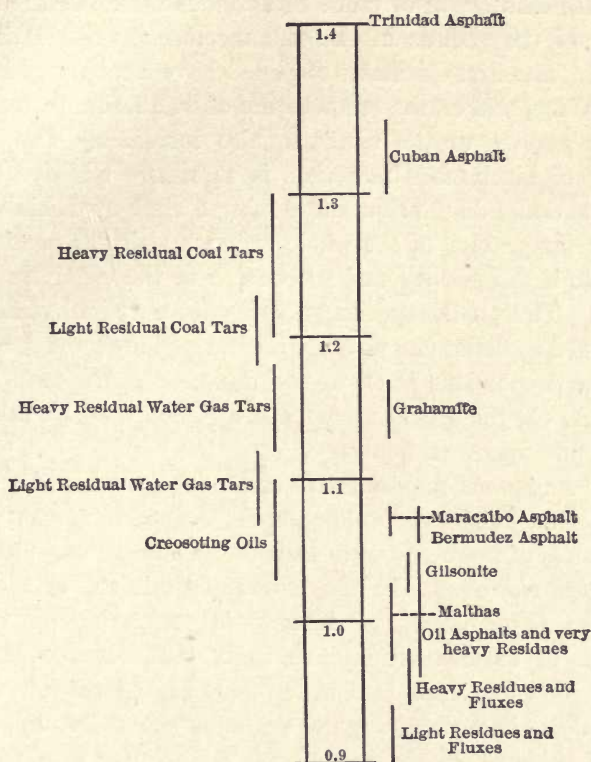


FIG. 8. Specific-Gravity Chart for Bituminous Materials

source. For this reason it is particularly valuable in the examination of a number of samples from a given shipment or lot of material before making a complete analysis of a composite sample. Thus, individual samples from a given lot of material will almost invariably be of uniform character if they have the same specific gravity and consistency test values.

In general, bituminous materials tend to classify themselves

according to specific gravity as illustrated by Fig. 8. The pure bitumen of petroleum and asphalt products seldom exceeds 1.055 and of tars 1.18. A higher specific gravity, therefore, usually indicates the presence of mineral matter or heavy inorganic impurities, such as free carbon.

In the distillation of crude bituminous materials the specific gravity of the residue in the still increases as distillation progresses. The first distillates obtained have a relatively low specific gravity, and as the temperature of distillation increases the specific gravity of the distillate also increases. The specific gravity of bituminous materials is so nearly additive that in both fractional distillation and fluxing a close approximation of the specific gravity of the whole or any particular portion may be made if the gravity and proportion of the other factors are known. Thus, if the specific gravities of an asphalt cement and the refined asphalt from which it is produced are known, together with the proportions of R. A. and flux used in the manufacture of the A. C., the specific gravity of the flux may be calculated with a fair degree of accuracy.

As bituminous materials are commonly purchased and used upon both a weight and volume basis, the specific-gravity determination is of frequent use in translating volume units to weight units, and *vice versa*. In the laboratory this use of the determination is of particular interest in comparing the bitumen contents of various bituminous aggregates. Moreover, in the examination of compressed bituminous aggregates it is of very material use in determining the degree of compression or percentage of voids which are present. It is also used in determining the coefficient of expansion of bituminous materials.

#### COEFFICIENT OF EXPANSION

##### *Basis of Determination*

The coefficient of volume or cubical expansion of any substance is the ratio between the increase in volume which it undergoes when its temperature is raised one degree and its original volume. The original or unit volume is set at some



standard temperature. In the case of bituminous materials, normal temperature,  $25^{\circ}\text{C.}$ , is convenient to use, although, for the same reason that the specific-gravity determination may be required at  $15.5^{\circ}\text{C.}$ , this temperature is also sometimes taken as standard. The coefficient of expansion may be expressed either in terms of the Centigrade or Fahrenheit scale. If  $K$  represents the coefficient of expansion, then  $K^{\circ}\text{C.} = \frac{9K^{\circ}\text{F.}}{5}$  and  $K^{\circ}\text{F.} = \frac{5K^{\circ}\text{C.}}{9}$ . The coefficient of expansion of most materials varies slightly at different temperatures, and for this reason the volume change which is undergone between comparatively wide ranges of temperature is divided by the number of degrees of temperature and the average coefficient of expansion calculated. It is the average coefficient of expansion of bituminous materials which is of most interest from a practical standpoint. As a material expands under the action of heat, its specific gravity becomes less. It is thus possible to calculate  $K$  from volume measurements at different temperatures or from specific-gravity determinations at different temperatures.

### *Specific-Gravity Method*

In this determination it is usually most convenient to use the pycnometer method of determining the specific gravity of the material at normal temperature. The weight of the water content of the pycnometer at any desired elevated temperature is thus obtained and another specific-gravity determination made of the material at this elevated temperature as compared with water at the same temperature. From the known  $K$  of water, which is about 0.0002 per  $^{\circ}\text{C.}$ , the specific gravity of the material at the elevated temperature is then determined as compared with water at normal temperature, as follows:

$$\text{Sp. Gr. } X^{\circ}/25^{\circ}\text{C.} = \frac{\text{Sp. Gr. } X^{\circ}/X^{\circ}\text{C.}}{1 + (X^{\circ} - 25^{\circ}) \times 0.0002}.$$

The coefficient of expansion of the material is then determined by means of the following formula, where  $G$  represents the spe-

cific gravity at normal temperature and  $G^1$  the specific gravity on the same basis at the observed temperature.

$$K = \frac{G - G^1}{G^1 (t^1 - t)}.$$

### *Value of Determination*

The practical value of the coefficient of expansion determination lies in its application to volume changes which take place upon heating bituminous materials, especially where the material is purchased or used upon a volume basis, which is often the case. In the vicinity of refineries, bituminous materials for hot-surface application or bituminous macadam construction are not uncommonly delivered at the site of work in tank wagons or tank cars at a temperature ready to apply. As the purchase price and rate of application are based upon volumes at normal temperature or at  $15.5^\circ \text{C.}$ , it is necessary to know the coefficient of expansion of the material in order to compute the volume at such temperature. In some cases an arbitrary figure is mutually agreed upon. Thus, for residual petroleum the coefficient of expansion is often assumed as 0.0004 per  $^\circ \text{F.}$  (0.00072 per  $^\circ \text{C.}$ ), and in measuring hot oils a deduction of 0.4 per cent is made for every  $10^\circ \text{F.}$  above  $60^\circ \text{F.}$  ( $15.5^\circ \text{C.}$ ), which is commonly taken as standard temperature. The general formulas for finding volumes when  $K$  is known are as follows:

$$V = \frac{V^1}{K (t^1 - t) + 1}$$

$$V^1 = V [K (t^1 - t) + 1].$$

Comparatively little data are to be had relative to the coefficient of expansion of the various bituminous road and paving materials. In general, however, for a given type of material the coefficient of expansion decreases as the specific gravity increases. Thus, the  $K$  of a crude petroleum or tar is greater than that of its residue and less than that of its distillates. From the data that are available, it appears that the following values of  $K$  are reasonable averages for the types of materials listed. These

figures are, however, only approximate, and in certain cases are subject to considerable variation.

#### APPROXIMATE COEFFICIENTS OF EXPANSION OF BITUMINOUS MATERIALS

Material	$K$ per ° C.	$K$ per ° F.
Gasoline . . . . .	0.00090	0.00050
Creosoting oils . . . . .	.00080	.00044
Fluid residual petroleums . . . . .	.00070	.00039
Fluid tars . . . . .	.00060	.00033
Asphalt cements . . . . .	.00055	.00030
Heavy refined tars . . . . .	.00055	.00030

## CONSISTENCY TESTS

### VISCOSITY

#### *Equipment:*

- 1 Engler viscosimeter complete with thermometers, burner, and rubber tubing.
- 1 100-cubic centimeter cylindrical glass graduate.
- 1 stop-watch.

*Method.*—The viscosity of fluid bituminous road materials is determined at any suitable temperature, most commonly by means of the Engler viscosimeter. This apparatus is shown in Fig. 9, and may be described as follows: *a* is a brass vessel for holding the material to be tested, and may be closed by the cover, *b*. To the conical bottom of *a* is fitted a conical outflow tube, *c*, exactly 20 millimeters long, with a diameter at the top of 2.9 millimeters and at the bottom of 2.8 millimeters. This tube can be closed and opened by the pointed hardwood stopper, *d*. Pointed metal projections are placed on the inside of *a* at equal distances from the bottom and serve for measuring the charge of material, which is 240 cubic centimeters. The thermometer *e* is used to ascertain the temperature of the material to be tested. The vessel, *a*, is surrounded by a brass jacket, *f*, which holds the material used as a heating bath, either water or cottonseed-oil, according to the temperature at which the test is to be made. A tripod, *g*, serves as a support for the apparatus, and also carries a ring burner, *h*, by means of which the bath



is directly heated. The measuring cylinder of 100 cubic centimeters capacity, which is sufficiently accurate for work with road materials, is placed directly under the outflow tube.

As viscosity determinations are frequently compared with that of water at  $25^{\circ}\text{C.}$ , the apparatus should be previously calibrated as follows: The cup and outlet tube should first be scrupulously cleaned. A piece of soft tissue paper is convenient for

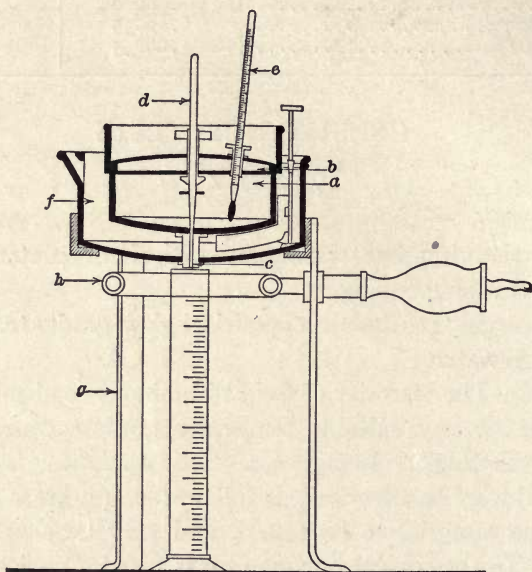


FIG. 9. Engler Viscosimeter

cleaning the latter. The stopper is then inserted in the tube and the cup filled with water at  $25^{\circ}\text{C.}$  to the top of the projections. The measuring cylinder should be placed directly under the outflow tube so that the material, upon flowing out, will not touch the sides, and the stopper may then be removed. The time required both for 50 and 100 cubic centimeters to run out should be ascertained by means of a stop-watch, and the results so obtained should be checked a number of times. The time required for 50 cubic centimeters of water should be about 11 seconds and for 100 cubic centimeters about 22.8 seconds.

Bituminous road materials are tested in the same manner as water, and the temperature at which the test is made is controlled by the bath. The material should be brought to the desired temperature and maintained there for at least three minutes before making the test. The results are expressed as so-called specific viscosity compared with water at 25° C., as follows:

$$\left. \begin{array}{l} \text{Specific vis-} \\ \text{cosity at } a^{\circ} \text{C.} \end{array} \right\} = \frac{\text{seconds for passage of given volume at } a^{\circ} \text{C.}}{\text{seconds for passage of same volume of water at } 25^{\circ} \text{C.}}$$

It should be noted that this formula does not give the actual specific viscosity, as it does not take into account the specific gravity of the material under examination. The method does, however, tend to correct and place upon the same basis different instruments.

For all thin, fluid bituminous road materials the specific viscosity is determined at 25° C. with 50 or 100 cubic centimeters. Viscous fluid products are run at 40° C. or 50° C. with 50 cubic centimeters, and very viscous products at 100° C. or over with 50 cubic centimeters.

### *Value of Determination*

While the Engler viscosimeter is far from ideal for testing the wide range of liquid bituminous materials used in highway engineering, it appears to be the most generally satisfactory instrument for this purpose that has yet been devised. Unfortunately, it is not well adapted for determining the specific viscosity of many bituminous materials at 25° C. As, for the different types of materials, viscosity at a higher temperature is no definite measure of their viscosity at 25° C., it is evident that a satisfactory comparison of their specific viscosity at normal temperature cannot be obtained by this means. As a means of such comparison the use of the instrument at higher temperatures is, therefore, limited to materials of the same type. The interpretation of the test, except with reference to identification and control of a given type and grade of material, is somewhat complicated. If, however, it is desired to apply a

bituminous material at a given temperature, as, for instance, when it is to be heated in a tank car by means of steam, a determination of its viscosity at that temperature is often of value. It is impossible to state in a general way just what the viscosity of a material for surface application should be during application, as this is largely dependent upon the type of distributor, its spraying nozzles, and its speed control. It is a fact, however, that many materials sold for cold-surface application are entirely too viscous at normal temperatures for uniform distribution at the proper rate of distribution, but what may be a too high viscosity for one type of material may not be too high for another type. As a measure of consistency at normal temperatures, it would appear advisable to limit the use of the Engler viscosimeter to those materials intended for cold-surface application, and where possible to utilize some other consistency test for indicating the consistency at normal temperature of those materials which have to be heated before application. In fact, a viscosity determination is seldom made upon a material whose consistency at normal temperature may be made with some other consistency test.

In general, at normal temperatures, fluid distillates show a much lower specific viscosity than do fluid petroleum or tar residuums. Crude and dehydrated petroleums and tars vary greatly in viscosity. Among the petroleums, however, those of the asphaltic type are usually much more viscous than the paraffin or semi-asphaltic type. Among the tars, the viscosity of crude water-gas tar is lowest and gas-house coal tars from horizontal retorts the highest. For a given type of fluid residual petroleum or tar produced by fractional distillation, the specific viscosity increases with the specific gravity and decreases with the coefficient of expansion. As regards fluid residuums which are subjected to the blowing process, however, the viscosity may be greatly increased without markedly increasing the specific gravity of the residuum. The viscosity of all bituminous materials decreases as their temperature increases. In any bituminous material the presence of finely divided non-bituminous impurities held in suspension will often greatly increase the vis-



cosity of the material, although the actual viscosity of its pure bitumen may be low.

The viscosity test is largely used as a control test in the manufacture of liquid bituminous materials of interest in highway engineering.

## FLOAT TEST

*Equipment:*

- 1 aluminum float or saucer. (Fig. 10a.)
- 2 conical brass collars. (Fig. 10b.)
- 2 1-quart tin cups, seamless.
- 2 chemical thermometers reading from  $-10^{\circ}$  C. to  $110^{\circ}$  C.
- 1 iron tripod.
- 1 Bunsen burner and rubber tubing.
- 1 burette clamp and support.
- 1 large metal kitchen spoon.
- 1 steel spatula or kitchen knife.
- 1 brass plate about six inches square and one-eighth inch thick.
- 1 stop-watch.

*Method.*—The New York Testing Laboratory float apparatus consists of two parts, an aluminum float or saucer (Fig. 10a),

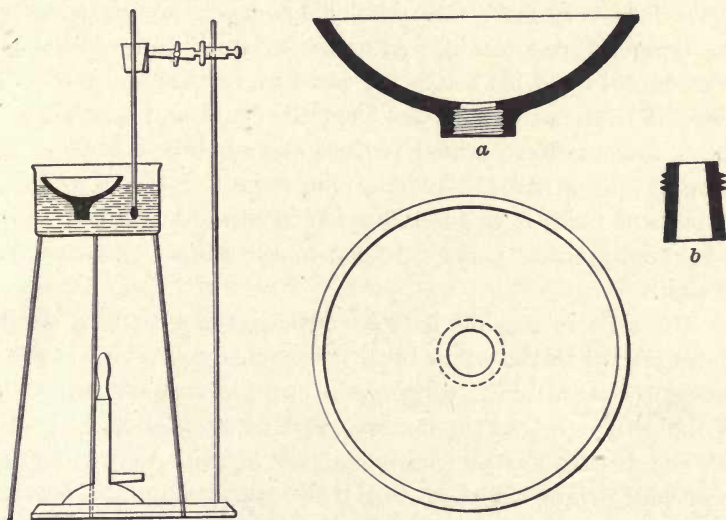


FIG. 10. New York Testing Laboratory Float Apparatus

and a conical brass collar (Fig. 10b). The two parts are made separately, so that one float may be used with a number of brass collars.

In making the test, the brass collar is placed with the small end down on the brass plate, which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate and then with mercury. A small quantity of the material to be tested is heated in the metal spoon until quite fluid, with care that it suffers no appreciable loss by volatilization and that it is kept free from air-bubbles. It is then poured into the collar in a thin stream until slightly more than level with the top. The surplus may be removed, after the material has cooled to room temperature, by means of a spatula or steel knife which has been slightly heated. The collar and plate are then placed in one of the tin cups containing ice water maintained at  $5^{\circ}\text{C}$ ., and left in this bath for at least 15 minutes. Meanwhile the other cup is filled about three-fourths full of water and placed on the tripod, and the water is heated to any desired temperature at which the test is to be made. This temperature should be accurately maintained, and should at no time throughout the entire test be allowed to vary more than one-half a degree Centigrade from the temperature selected. After the material to be tested has been kept in the ice water for at least 15 minutes, the collar with its contents is removed from the plate and screwed into the aluminum float, which is then immediately floated in the warmed bath. As the plug of bituminous material becomes warm and fluid, it is gradually forced upward and out of the collar until water gains entrance to the saucer and causes it to sink.

The time in seconds between placing the apparatus on the water and when the water breaks through the bitumen is determined by means of a stop-watch and is taken as a measure of the consistency of the material under examination.

The float test is preferably made at  $50^{\circ}\text{C}$ . Very soft products which show a test at this temperature of only a few seconds, however, may be advantageously tested at a lower tem-

perature, such as 32° C. If the test at 50° C. requires over four or five minutes, it is sometimes made at 65° C., and if the melting point of the material is high, the test is run at 100° C.

### *Value of Test*

The float test has sometimes been called a viscosity test. In some respects it is a measure of the resistance to flow possessed by the material, but it differs from the average viscosity test mainly in the fact that the temperature of the material itself is constantly changing throughout the test. It cannot be used with very fluid materials, but for the very viscous fluids which become almost semisolid at the temperature of ice water and for many normally semisolid products it serves as a very satisfactory measure of consistency at normal temperature, although the test itself is conducted at a higher temperature. Providing the temperature of the test is the same, it serves reasonably well as a basis of comparing the consistency at normal temperature of both straight residual and fluxed products of the same type. It has the distinct advantage over the viscosity test of being less susceptible to the influence of inert, finely divided suspended material which may be present, because of the comparatively large orifice of the collar. For this reason it more nearly represents the consistency of the actual bitumen, and is particularly well adapted for determining the consistency of tars, irrespective of their free carbon content. In this connection it is used to a considerable extent as a control test in the manufacture of heavy residual tars. It is not as well adapted for testing the semisolid blown-oil products which conduct heat so slowly that only the surface in contact with the collar becomes fluid before the entire plug of semisolid material is forced out. For a given type of residual material the float test at a given temperature decreases with the specific gravity. For any material it naturally decreases as the temperature of the test is increased. For fluid products which harden rapidly upon exposure, such as cut-backs, the float test is useful as a means of determining the consistency of the residue from the volatiliza-



tion test, especially the soft residues which cannot well be subjected to the penetration test.

#### PENETRATION TEST

##### *Equipment:*

- 1 penetrometer complete, with a seconds pendulum or metronome. (Figs. 11 and 12.)
- 1 tin box, approximately 5.5 centimeters in diameter by 3.5 centimeters in height.
- 1 large metal kitchen spoon.
- 1 steel spatula or kitchen knife.
- 1 glass penetration dish, approximately 10 centimeters in diameter by 6 centimeters high.
- 1 enamelware dish, approximately 3 inches deep and 9 inches in diameter.
- 1 chemical thermometer reading from  $-10^{\circ}$  C. to  $110^{\circ}$  C.

*Method.*—The object of the penetration test is to ascertain the consistency of the material under examination by determining the distance a weighted needle will penetrate into it at a given temperature under known conditions of time and load. A standard needle is employed for this purpose, and this needle is usually weighted with 100 grams. The depth of penetration is most commonly determined upon the bitumen maintained at  $25^{\circ}$  C., while the load is applied for five seconds.

The standard needle is made from round, polished, annealed-steel drill-rod having a diameter of from 0.0405 to 0.0410 inches. The rod is tapered to a sharp point at one end, with the taper extending back one-fourth inch. It is then highly polished, tempered, and again polished with jewelers' rouge. The finished needle is from  $1\frac{3}{4}$  to 2 inches in length and exactly 0.040 inch in diameter. This needle gives the same results as the old standard No. 2 cambric needle, and possesses the advantage that it can be exactly duplicated and accurately described.

There are a number of penetration machines in common use which employ the same standards of test, and therefore give practically equivalent results. Among them may be mentioned the Dow, the New York Testing Laboratory, and the Hum-

boldt penetrometers. The Dow machine, shown in Fig. 11, consists of a standard needle, *a*, inserted in a short brass rod, which is held in the aluminum rod, *b*, by a binding screw. The aluminum rod is secured in a framework so weighted and balanced that, when it is supported on the point of the needle, the framework and rod will stand in an upright position, allowing

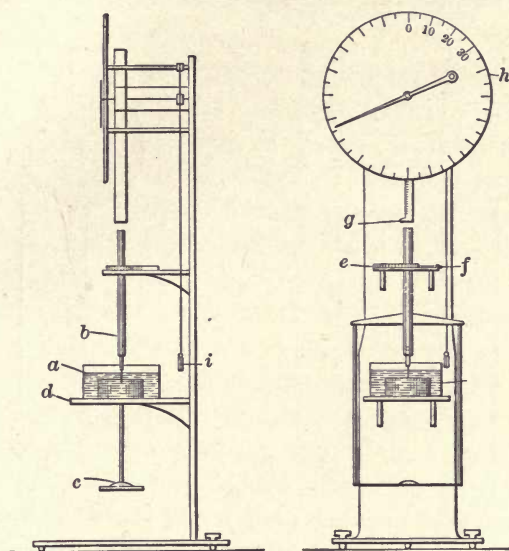


FIG. 11. Dow Penetration Machine

the needle to penetrate perpendicularly without the aid of a support.

The frame, aluminum rod, and needle weigh 100 grams with the weight *c* on the bottom of the frame, while without the weight they weigh 50 grams. Fig. 11 shows the needle and weighted frame, together with side and front views of the entire apparatus, put together and ready for making a penetration. The shelf for the sample is marked *d*; *e* is the clamp to hold the aluminum rod until it is desired to make a test, and *f* is a button which, when pressed, opens the clamp. By turning this button while the clamp is being held open, it will lock and keep the clamp from closing until unlocked. The device for measuring

the distance penetrated by the needle consists of a rack, with a foot, *g*. The movement of this rack turns a pinion, to which is attached the hand which indicates on the dial, *h*, the vertical distance covered by the rack. One division of the dial corresponds to a movement of 0.01 centimeter by the rack. The rack may be raised or lowered by moving the counterweight, *i*, up or down. The tin box containing the sample to be tested is

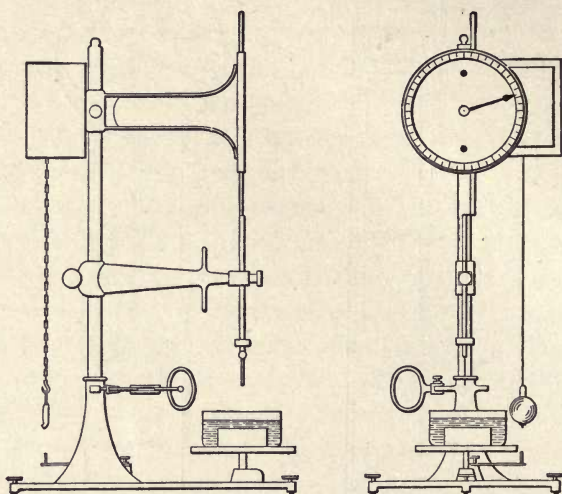


FIG. 12. New York Testing Laboratory Penetrometer

marked *k*; this is submerged in water contained in the glass cup in order to maintain a constant temperature.

Another type of machine known as the New York Testing Laboratory penetrometer, based upon the same general principle and using the same standards, is shown in Fig. 12. Both machines give practically the same results if operated under the same conditions, and it is therefore considered unnecessary to include a description of the latter.

A cup suitable for holding the box containing the test sample during penetration is conveniently made from a glass crystallizing dish 10 centimeters in diameter, with straight sides about 6 centimeters high. Three right triangles with right-angle sides, 1 and 5 centimeters, respectively, are cut from 1/16-inch sheet



metal. Some solid bitumen is melted in the bottom of the dish, forming a layer about  $\frac{1}{8}$  inch thick, into which the triangles are placed, resting on the side five centimeters long. Their apexes should meet at the center, with their short sides dividing the circumference of the dish into three equal arcs. When the bitumen has hardened, the triangles give a firm support for circular boxes, and the possibility of any rocking motion and consequent faulty results is avoided.

The penetration test is made as follows: A sample of the material to be tested is first warmed sufficiently to flow, and poured into the box.\* The box and contents, after cooling for one hour at room temperature, are immersed in water maintained at the temperature at which the test is to be made, and allowed to remain immersed for one hour. The sample in the tin box should now be placed in the glass cup and removed in it, covered with as much water as convenient without spilling, to the shelf *d*. The brass rod with the needle is inserted into *b* and secured by tightening the binding screw. The rod is lowered until the point of the needle almost touches the surface of the sample; then by grasping the frame with both hands it is cautiously pulled down until the needle just comes in contact with the surface of the sample. This can be seen best by having a light so situated that, upon looking through the sides of the glass cup, the needle will be reflected from the surface of the sample. After thus setting the needle, the counterweight is slowly raised until the foot of the rack rests on the head of the rod and a reading of the dial taken. The clamp is then opened wide by pressing the button and held in this position for exactly five seconds, as determined by the pendulum or metronome. The clamp is then released, the rack lowered until it rests on the rod, and the difference between the first and second readings of the dial in hundredths of a centimeter is taken as the distance penetrated by the needle.

Owing to the susceptibility of certain bitumens to slight changes in temperature, the water bath should be accurately

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\* American Can Company's Gill style ointment-box, deep pattern, 3-oz. capacity, meets the requirements.

maintained at the desired temperature, both before and during the test to within  $0.1^{\circ}\text{C}.$ , and, when the room temperature differs greatly from that of the bath, the water in the glass cup should be renewed after each test. An average of from three to five tests, which should not differ more than four points between maximum and minimum, is taken as the penetration of the sample. The tests should be made at points on the surface of the sample not less than one centimeter from the side of the container and not less than one centimeter apart.

The needle should be removed and thoroughly cleaned by wiping with a dry cloth, after which it is ready for another test. The point of the needle should be examined from time to time with a magnifying glass to see that it is not injured in any way. If it is found defective it may be removed by heating the brass rod and withdrawing with pliers. A new needle may then be inserted in the heated brass rod. and held firmly in place by a drop of soft solder.

While the standard conditions under which this test is made call for a 100-gram load applied for five seconds on the material maintained at a temperature of  $25^{\circ}\text{C}.$ , it is sometimes desirable, when very soft materials are tested, to make the test with a 50-gram weight. In order to ascertain how susceptible a material may be to temperature changes, tests may be made at any other desired temperatures, preferably  $0^{\circ}\text{C}.$  with a 200-gram weight for one minute, and at  $46^{\circ}\text{C}.$  with a 50-gram weight for five seconds. When the test is made at  $0^{\circ}\text{C}.$  the sample should be immersed in brine.

In all cases the results of tests should be reported in hundredths of a centimeter, as follows, showing all the conditions in order that no misinterpretation of results may occur:

Penetration (— seconds. — grams at —  $^{\circ}\text{C}.$ ) = —.

### *Value of Test*

The penetration test is most commonly used in connection with asphalts and asphalt cements, although it is sometimes used in testing the harder-tar residuums. For the manufacture

of the former type of materials it serves as a control test. These materials are commonly graded by their penetration at  $25^{\circ}\text{C}.$ , and in all cases where the term "penetration" is used without other qualification it is understood that the temperature of  $25^{\circ}\text{C}.$  is implied and that the test is made with a standard needle under a load of 100 grams applied for five seconds. The test is not as useful as a means of control or in grading the tar residuums used in highway engineering for the reason that most of these residuums are quite soft, and, as their surface tension is very high, they press out or deform under the loaded needle much more than do the asphalt cements commonly used. The true penetration is not, therefore, as accurately recorded, the result obtained representing to a very considerable extent the measure of surface deformation rather than actual penetration. In general, the more susceptible the bituminous material is to temperature changes the greater its surface deformation under the loaded needle. Susceptibility to temperature changes may be determined by means of the penetration test made at the temperatures of  $0^{\circ}$  or  $4^{\circ}$ ,  $25^{\circ}$ , and  $46^{\circ}\text{C}.$ , which serve as approximate minimum average and maximum temperatures of the pavement proper in which the material may be incorporated.

In the manufacture of bituminous materials the semisolid residues produced by distillation become harder and harder as distillation progresses, or, in other words, the penetration of these residues decreases with distillation. For a given type of residue the penetration, therefore, decreases as the specific gravity increases. This relation is not, however, so marked in the case of blown products, which may decrease markedly in penetration with very slight change in specific gravity as the blowing process progresses. In comparing different types of materials there is no definite relation between penetration and specific gravity. Thus an asphalt cement of a given penetration produced by straight distillation of a California petroleum will usually show a much lower specific gravity than will one of the same penetration from a Mexican petroleum or by fluxing a native asphalt. Highly blown asphalt cements may usually be identified by a very low specific gravity as compared



with residual and fluxed native asphalts of the same penetration. Thus the penetration test, while practically valueless alone as a means of identification, owing to the fact that almost any type of material can be manufactured at any desired penetration, may be a valuable aid to identification when considered in connection with the specific gravity of the material.

Besides being applied to the original material the penetration test is ordinarily made where possible upon the residue from the volatilization test to determine what changes in consistency have been produced by volatilization. It is also of great importance in the plant inspection of asphalt cements prepared at the paving plant by fluxing refined asphalts. When made at various temperatures it may be used to determine the susceptibility of a material to temperature changes.

#### MELTING OR SOFTENING-POINT TESTS

##### *Equipment:*

##### *Cube Method*

- 1 iron tripod.
- 1 Bunsen burner and rubber tubing.
- 1 piece of wire gauze 10 centimeters square.
- 1 800-cubic centimeter Jena glass beaker, low form. (Fig. 13a.)
- 1 400-cubic centimeter Jena glass beaker, tall without lip. (Fig. 13b.)
- 1 iron ring support (ring 7.5 centimeters in diameter) and burette clamp. (Fig. 13c.)
- 1 metal cover. (Fig. 13d.)
- 1 object glass.
- 1 piece of wire (No. 12 Brown & Sharpe gauge) 20 centimeters in length, bent. (Fig. 13e.)
- 1 chemical thermometer reading from 0° C. to 250° C.
- 1 cubical brass mold. (Fig. 13f.)
- 1 brass plate about six inches square and  $\frac{1}{8}$ " thick.
- 1 large metal kitchen spoon.
- 1 steel spatula or kitchen knife.

The material under examination is first melted in the spoon by the gentle application of heat until sufficiently fluid to pour readily. Care must be taken that it suffers no appreciable loss by volatilization. It is then poured into the  $\frac{1}{2}$ -inch brass cubical mold, which has been amalgamated with mercury and which is placed on an amalgamated brass plate. The brass may be amalgamated by washing it first with a dilute solution of mercuric chloride or nitrate, after which the mercury is

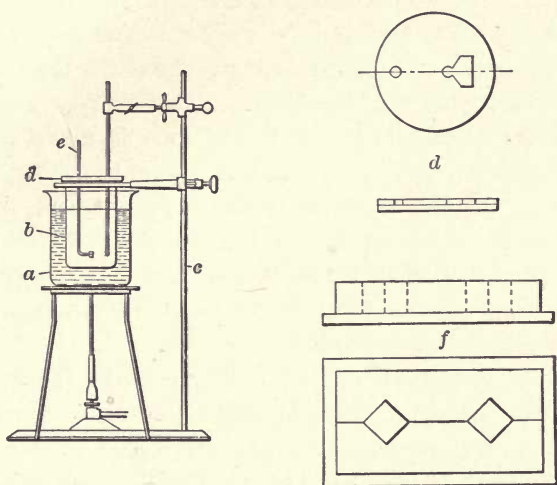


FIG. 13. Melting-Point Apparatus—Cube Method

rubbed into the surface. By this means the bitumen is, to a considerable extent, prevented from sticking to the sides of the mold. The hot material should slightly more than fill the mold and, when cooled, the excess may be cut off with a hot spatula or knife.

After cooling to room temperature, the mold is placed in a bath maintained at  $25^{\circ}\text{C}$ . for one half-hour. The cube is then removed and fastened upon the lower arm of a No. 12 wire (Brown & Sharpe gauge), bent at right angles and suspended beside a thermometer in a covered Jena glass beaker of 400 cubic centimeters capacity, which is placed in a water bath, or, for high temperatures, a cottonseed-oil bath. The wire

should be passed through the center of two opposite faces of the cube, which is suspended with its base 1 inch above the bottom of the beaker. The water or oil bath consists of an 800-cubic centimeter low-form Jena glass beaker suitably mounted for the application of heat from below. The beaker in which the cube is suspended is of the tall-form Jena type without lip. The metal cover has two openings as shown in Fig. 13*d*. A cork, through which passes the upper arm of the wire, is inserted in one hole and the thermometer in the other. The bulb of the thermometer should be just level with the cube and at an equal distance from the side of the beaker. In order that a reading of the thermometer may be made, if necessary, at the point which passes through the cover, the hole is made triangular in shape and covered with an ordinary object glass through which the stem of the thermometer may be seen. Readings made through this glass should be corrected for the angle of observation, which may be made constant by always sighting from the front edge of the opening at any given point on the stem of the thermometer below the cover.

After the test specimen has been placed in the apparatus, the liquid in the outer vessel is heated in such a manner that the thermometer registers an increase of 5° C. per minute. The temperature at which the bitumen touches a piece of paper placed in the bottom of the beaker is taken as the melting point. Determinations made in the manner described should not vary more than two degrees for different tests of the same material. At the beginning of this test the temperature of both bitumen and bath should be approximately 25° C. For tar products the test is sometimes run with the cube suspended directly in water, in which case only the smaller beaker is required.

#### *Ring and Ball Method*

##### *Equipment:*

- 1 iron tripod.
- 1 Bunsen burner and rubber tubing.
- 1 piece wire gauze 10 centimeters square with asbestos center.



- 1 600-cubic centimeter beaker.
- 1 ring attached to heavy wire.
- 1 steel ball.
- 1 cork.
- 1 iron stand with burette clamp.
- 1 chemical thermometer reading from 0° C. to 250° C.
- 1 brass plate about six inches square and  $\frac{1}{8}$ " thick.
- 1 large metal kitchen spoon.
- 1 steel spatula or kitchen knife.

*Method.*—This method for other than tar products has been recommended to the American Society for Testing Materials for adoption as standard by Committee D-4. It is, however, here described somewhat more in detail than in the Committee's recommendations.

The mold consists of a brass ring  $\frac{5}{8}$  inch in diameter and  $\frac{1}{4}$  inch thick, with a wall  $\frac{3}{32}$  inch thick. The ring is fastened to a stout wire in a horizontal position at right angles to the wire. A steel ball  $\frac{3}{8}$  inch in diameter and weighing between 3.45 and 3.50 grams is also required. When ready for test the apparatus is set up as shown in Fig. 14 upon the metal tripod, the ring being suspended in a horizontal position inside and one inch above the bottom of the beaker, which should contain approximately 400 cubic centimeters of water at 5° C.

The material under examination is first melted in the spoon by the gentle application of heat until sufficiently fluid to pour readily. Care must be taken that it suffers no appreciable loss by volatilization. It is then poured into the ring, which is placed upon an amalga-

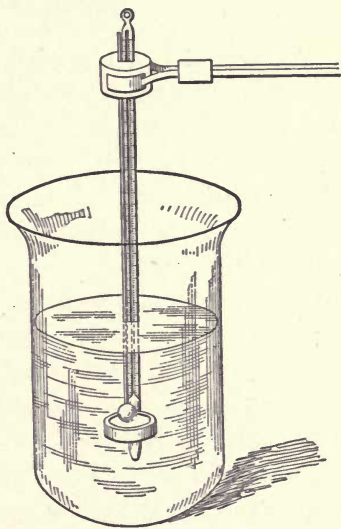


FIG. 14. Melting-Point Apparatus  
—Ring and Ball Method

mated brass plate. The hot material should slightly more than fill the ring, and when cooled the excess may be cut off with a hot spatula or knife. The ball is next placed upon the surface of the material in the center of the ring and the whole suspended in the beaker as above described, with the thermometer bulb within  $\frac{1}{2}$  inch of the sample and at the same level. Heat is then applied uniformly to the bottom of the beaker in quantity sufficient to raise the temperature of the water  $5^{\circ}$  C. per minute. The temperature is recorded at the start of the test and every minute thereafter, as the rate of heating is a very important factor. The melting or softening point is that temperature at which the material first touches the bottom of the beaker. Successive tests should average within  $3^{\circ}$  C. For melting points above  $95^{\circ}$  C. a glycerine bath should be used instead of water.

### *Value of Melting-Point Test*

As bituminous materials are not definite individual compounds and as those which are ordinarily termed solids are not true solids but practically solid solutions, it follows that they can have no true melting point. In other words, bitumen which may be hard and brittle at normal temperature will, when heated, gradually become softer and softer until it flows readily and no critical temperature can be observed for its change from apparent solid to liquid form. Any method of determining the so-called melting point of bituminous materials is therefore purely arbitrary and gives purely arbitrary results. For this reason different methods give different results with the same material, and in reporting results or preparing specifications it is therefore necessary to indicate the method used or to be used. No absolutely definite relation applicable to bituminous materials in general has as yet been found to exist between the cube method and the ring and ball method.

A melting-point test may be made upon any type of bitumen which is sufficiently solid to hold its shape for some time under conditions maintained at the beginning of the test. In

the manufacture of tar pitches and blown petroleums or asphalts it is often used as a control test, and such materials are sometimes graded by their melting point. As different manufacturers use different methods, however, a stated melting point has no particular significance unless the method is known. In general, as the distillation of a bituminous material progresses, the melting point of the semisolid and solid residues increases. For straight-distilled products of a given type, therefore, the melting point increases with an increase in specific gravity and an increase in float test or a decrease in penetration at any given temperature. If, however, the blowing process is used, particularly with petroleum products, an increase in melting point may be obtained with a very slight increase in specific gravity and decrease in penetration as compared with the accompanying changes in specific gravity and penetration had the product been distilled to the same melting point. By properly manipulating the distilling and blowing processes, various combinations of melting point and penetration may be secured from the same original material. For a given penetration at normal temperature, high melting-point materials are as a rule less susceptible to the ordinary temperature changes than are low melting-point materials.

In connection with the specific gravity and penetration tests the melting-point test may serve as a means not only of identifying a material but also of ascertaining its method of manufacture. Besides being applied to the original material the melting-point test may be made upon the residue from the distillation test of tars, for the purpose of identifying cut-back hard pitches. It is seldom made upon the residue from the volatilization test.

## DUCTILITY

*Equipment:*

- 1 ductility machine complete with briquet molds.
- 1 large metal kitchen spoon.
- 1 steel spatula or kitchen knife.
- 1 brass plate about 6 inches square and  $\frac{1}{8}$  inch thick.
- 1 chemical thermometer reading from  $-10^{\circ}$  to  $110^{\circ}$  C.



*Method.*—Like the penetration test, there are a number of machines in common use for determining the ductility of bituminous materials which employ the same standards, and therefore give practically equivalent results. The test as ordinarily applied to asphalt cements was devised by Dow, whose machine is perhaps the best known. Other types in common use are the Kirschbraun machine and the Chew machine, which latter is shown in Fig. 15. In all of these machines a test specimen of the same standard shape and size is tested by pulling it apart

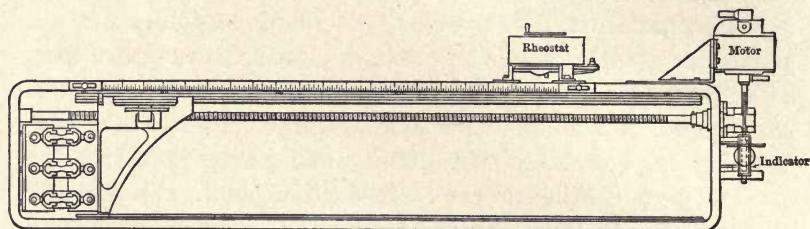


FIG. 15. Chew Ductility Machine

at a given rate and noting the distance that it stretches before breaking. In some cases the pulling is accomplished by means of a gear operated by a hand wheel, but a motor drive such as shown in the figure is to be preferred, as it insures greater uniformity in the rate of pull. The Chew machine also has an automatic device for indicating the rate of pull.

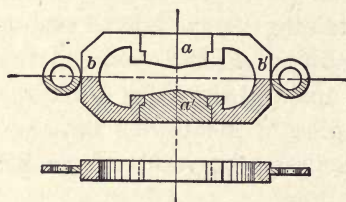


FIG. 16. Briquet Mold for Ductility Test

The test substantially as recommended in 1915 by "The Special Committee on Road Materials of The American Society of Civil Engineers" is as follows:

The briquet mold shown in Fig. 16 is made of brass and consists of four parts, two end clips (*a a*) and two side pieces (*b b*). When these parts are properly fitted together they should cast a briquet 1 centimeter in thickness throughout

its entire length. The distance between the end clips should be 3 centimeters, the width at the mouth of the clips should be 2 centimeters, and the width at the minimum cross-section, half-way between the clips, should be 1 centimeter.

When casting a briquet the inside faces of the two side pieces are first thoroughly amalgamated with mercury. The mold is then put together and placed upon an amalgamated brass plate. The material to be tested is next heated in a metal spoon until quite fluid, with care that it suffers no appreciable loss by volatilization. It is then poured into the mold until slightly more than level with the top. The surplus may be removed, after the material has cooled to room temperature, by means of a spatula or steel knife which has been slightly heated. The two side pieces of the mold are next removed, leaving the briquet of material held at each end by the ends of the mold, which now play the part of clips. In case the material tends to adhere to the sides of the mold their removal may be facilitated by immersing the material for a few moments in cold water.

The briquet with clips attached is then placed in water which is carefully maintained at  $25^{\circ}$  C. and allowed to remain for not less than thirty minutes. It is then transferred to the ductility machine which is filled with water at the same temperature. This machine consists of a rectangular water-tight box, having a movable block working on a worm gear from left to right. The left clip is held rigid by placing its ring over a short metal peg provided for this purpose; the right clip is placed over a similar rigid peg on the movable block. The movable block is provided with a pointer which moves along a centimeter scale. Before starting the test, the centimeter scale is adjusted to the pointer at zero. Power is then applied by the worm gear pulling from left to right at the uniform rate of 5 centimeters per minute. The distance, in centimeters, registered by the pointer on the scale at the time of rupture of the thread of bitumen is taken as the ductility of the material.

Most ductility machines are equipped for testing three

briquets simultaneously. The test is almost invariably made at 25° C., although 4° C. has also been recommended.

### *Value of Test*

As ordinarily applied, the ductility test is of little value other than as a means of identification when considered in connection with certain other tests and for control in the manufacture of certain types of bituminous materials, especially when the blowing process is used. The blowing process tends to materially reduce the ductility of an asphalt cement, and highly blown products almost invariably show a very low ductility.

For a given penetration at 25° C. those materials which are most susceptible to temperature changes are the most ductile at 25° C. As for a given type of material ductility decreases with penetration, it follows that most materials with a high ductility at 25° C. show a very low ductility at 4° C. Materials which are but slightly susceptible to temperature changes, such as blown asphalts, as a rule have a low ductility at 25° C., but as their penetration at 4° C. is not so greatly changed, neither is their ductility. If the ductility test is considered as a measure of the stretch of the bituminous material when incorporated in a pavement, which stretch may be caused by contraction of the pavement, it would appear that high ductility at 25° C. should in many cases be undesirable because it represents low ductility at the low temperatures which cause material contraction. As a matter of fact, it is more than doubtful if the test as conducted in any way represents the behavior of the comparatively thin films of material as they exist in a bituminous pavement. The theory has also been advanced that ductility is a measure of adhesiveness. Certainly those materials which appear to be most sticky at normal temperature show a high ductility at normal temperature, but, if the theory holds true, their adhesiveness falls off more markedly with decrease in temperature than does the initial adhesiveness of materials with a normally low ductility. Also, if this theory is unqualifiedly admitted, it would seem reasonable to conclude that the adhesiveness of an asphalt cement of, say, 60 penetra-



tion is less than that of the same type of asphalt cement of 150 penetration. The binding strength of the former is, however, known to be higher. From the above it is evident that correct interpretation of the ductility test, except as a means of identification and control, is no simple matter. In general, for a given type of bituminous material the ductility decreases as the specific gravity increases.

## HEAT TESTS

### FLASH AND BURNING POINTS

#### *Open-Cup Method*

##### *Equipment:*

- 1 open-cup oil tester with Bunsen burner. (Fig. 17.)
- 1 chemical thermometer  $0^{\circ}\text{C. to } 400^{\circ}\text{C.}$
- 1 piece of 6-millimeter glass tubing, 6 centimeters in length, one end of which has been drawn to a 1-millimeter opening. Soft rubber tubing for gas connections.

*Method.*—A number of open-cup oil testers have been devised which are similar in design and give practically equivalent results. This type is shown in Fig. 17. It consists of a brass oil cup, *a*, of about 100 cubic centimeters capacity. The outer vessel, *b*, serves as an air jacket. The thermometer, *c*, is suspended from the wire support, *d*, directly over the center of the cup so that its bulb is entirely covered with oil but does not touch the bottom of the cup. A testing flame is obtained from a jet of gas passed through a piece of glass tubing, and should be about 5 millimeters in length.

The test is made by first filling the oil cup with the material under examination to within

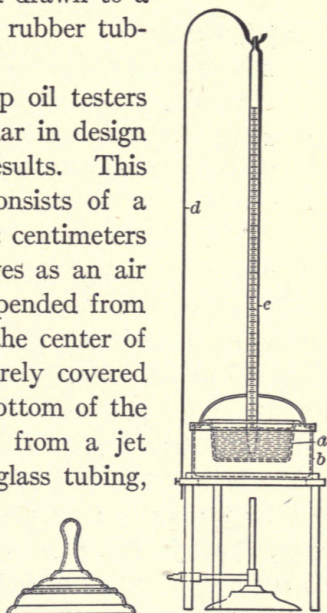


FIG. 17. Open-Cup Oil Tester

about 5 millimeters of the top. The Bunsen flame is then applied in such a manner that the temperature of the material in the cup is raised at the rate of  $5^{\circ}\text{C.}$  per minute. From time to time the testing flame is brought almost in contact with the surface of the oil. A distinct flicker or flash over the entire surface of the oil shows that the flash point is reached and the temperature at this point is taken. It will usually be found that the flash point as determined by the open-cup method is somewhat higher than by the closed-cup method for the same material.

The burning point of the material is obtained by continuing the test and noting that temperature at which it ignites and burns. The flame should then be extinguished by means of a metal cover supplied with the instrument. No attempt should be made to blow out the flame, for fear of scattering the burning oil.

#### *Closed-Cup Method*

##### *Equipment:*

- 1 New York State Board of Health oil tester with Bunsen burner. (Fig. 18.)
- 1 chemical thermometer reading from  $0^{\circ}\text{C.}$  to  $400^{\circ}\text{C.}$
- 1 piece of 6-millimeter glass tubing, 6 centimeters in length, one end of which has been drawn to a 1-millimeter opening. Soft rubber tubing for gas connection.
- 1 wire frame for holding thermometer.

*Method.*—While for all ordinary purposes the open-cup method of determining the flash and burning points of bituminous road materials is satisfactory, the closed-cup method is to be preferred where greater accuracy is required. This is particularly true for materials of a relatively low flash point.

The New York State Board of Health oil tester is ordinarily used for testing materials used in highway engineering. This tester, shown in Fig. 18, consists of a copper oil cup, *a*, of about 300 cubic centimeters capacity, which is heated in an oil bath, *b*, by a small Bunsen flame. The cup is provided with a glass cover, *c*, carrying a thermometer, *d*, and a hole, *e*, for inserting



the testing flame. The testing flame is obtained from a jet of gas passed through the piece of glass tubing and should be about 5 millimeters in length.

The flash test is made as follows: The oil cup should first be removed and the bath filled with cottonseed oil. The oil cup should be replaced and filled with the material to be tested to within 3 millimeters of the flange joining the cup and the vapor chamber above. The glass cover is then placed on the

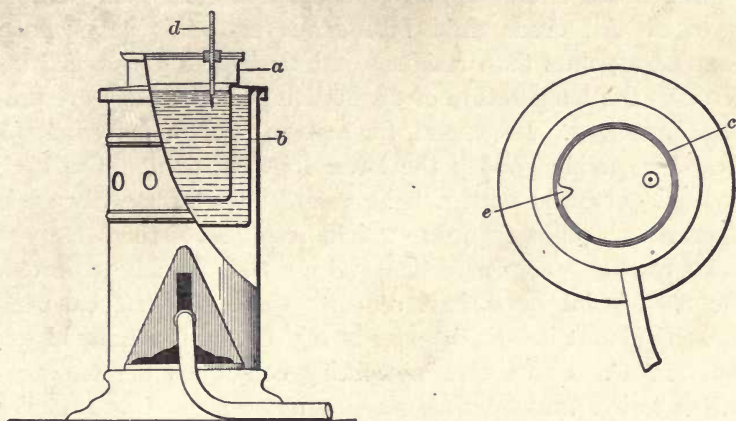


FIG. 18. New York State Board of Health Oil Tester

oil cup and the thermometer so adjusted that its bulb is just covered by the bituminous material. The Bunsen flame should be applied in such a manner that the temperature of the material in the cup is raised at the rate of  $5^{\circ}\text{C.}$  per minute. From time to time the testing flame is inserted in the opening in the cover to about half way between the surface of the material and the cover. The appearance of a faint bluish flame over the entire surface of the bitumen shows that the flash point has been reached and the temperature at this point is taken.

The burning point of the material may now be obtained by removing the glass cover and replacing the thermometer in a wire frame. The temperature is raised at the same rate and the material tested as before. The temperature at which the material ignites and burns is taken as the burning point.



At the conclusion of this test the flame should not be blown out, for danger of splashing the hot material. A metal cover or extinguisher should be employed for this purpose by placing it over the ignited material.

### *Value of Flash- and Burning-Point Determinations*

The flash and burning point determinations are of value as a quick means of differentiating heavy crude and fluid residual products. All crude fluid bituminous materials have much lower flash points than residuals, the flash points of which increase as the temperature of distillation at which they are produced increases. In general, for a given type of material, the lower its specific gravity the lower its flash point. Cut-back products produced with a light volatile flux necessarily show a lower flash point than do straight residuals of the same viscosity, float test, or penetration, and are thus quickly indicated. The flash point of asphalt cements which are not cut-back products almost invariably exceeds  $163^{\circ}\text{C}$ . and is usually higher than  $200^{\circ}\text{C}$ . Under certain working conditions the temperature at which a material flashes may be considered as a danger point, and in specifications the flash-point limit is often placed at a higher temperature than that to which it will be necessary to heat the material while being used. Sometimes the flash and burning points of a material are close together and in other cases they are quite far apart. The relation of flash to burning point is largely dependent upon the amount present in the material of that volatile constituent which first flashes. The flash and burning points of a mixture of hydrocarbons are seldom those of the most volatile constituents, as the presence of the heavier bodies tends to retard volatilization of the lighter. The flash and burning points of two or more constituents of a mixture are not additive, so that those of the mixture cannot be predetermined. The mixture should, however, show flash and burning points lying between those of the constituents. In reporting results and in specifying, the type of cup should be indicated, as the open cup usually gives considerably higher

results than the closed cup, in which the evolved vapors are more or less confined.

## VOLATILIZATION TEST

*Old Method**Equipment:*

- 1 constant-temperature hot-air oven with rubber tubing. (Fig. 19.)
- 1 thermo-regulator. (Fig. 19a.)
- 2 chemical thermometers reading from  $-10^{\circ}$  C. to  $250^{\circ}$  C.
- 2 tin boxes, 6 centimeters in diameter and 2 centimeters deep.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

*Method.*—The object of the volatilization test is to determine the percentage of loss which the material undergoes when a given weight of the material in a standard-sized container is subjected to a uniform temperature of  $163^{\circ}$  C. for five hours, and also to ascertain any changes in the character of the material due to such heating. The test as commonly made in connection with asphalts, fluxes, and asphalt cements, called for a 20-gram sample in a tin box 6 centimeters in diameter and 2 centimeters deep, but for a number of reasons a 50-gram sample in a box 3.5 centimeters deep, as called for in the new method, is to be preferred.

The oven shown in Fig. 19, known as the New York Testing Laboratory oven, is most commonly used, although any other form may be used that will give a uniform temperature throughout all parts where samples are placed. The oven is cylindrical in shape and consists of an inner compartment fitted with a circular, perforated metal shelf for holding the samples under test. This shelf should be covered with a heavy perforated sheet of asbestos to minimize the direct conduction of heat from the metal to the sample holders. A cylindrical outer compartment, equipped with a hinged cover at the top, is fitted over the inner compartment, leaving an air space between the

two. The oven is heated from below by means of a ring burner with perforations systematically placed so as to equalize the flames as nearly as possible. The top or cover of the oven contains a number of openings to allow air circulation or the insertion of thermometers and gas regulator. The oven is usually equipped with a fan or air stirrer below the shelf which may be operated from a pulley connected to a shaft which extends upward through the center opening in the top. Use of the fan during the test has, however, been generally discontinued. A gas regulator is inserted through one of the open-

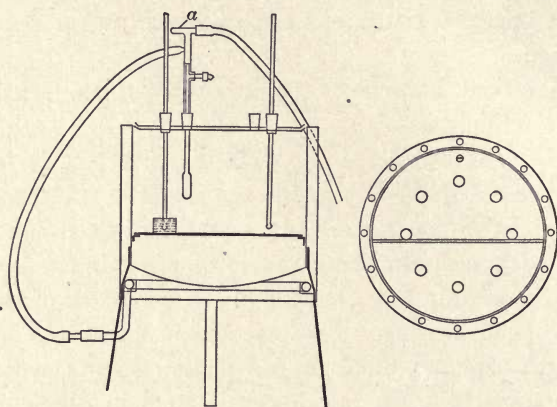


FIG. 19. New York Testing Laboratory Oven

ings in the top of the oven, and two thermometers are inserted through others. The bulb of one of the thermometers is immersed in a sample of some fluid, non-volatile bitumen, while the other is kept in air at the same level. The first thermometer serves to show the temperature of the samples during the test, while the latter gives prompt warning of any sudden changes in temperature due to irregularities in the gas pressure, etc.

Before making the test the interior of the oven should show a temperature of  $163^{\circ}\text{C}$ . as registered by the thermometer in air. The tin box is accurately weighed after carefully wiping with a towel to remove any grease or dirt. About 20 grams of the material to be tested are then placed in the box. The



material may first be weighed on a rough balance, if one is at hand, after which the accurate weight, which should not vary more than 0.2 gram from the specified amount, is obtained. It may be necessary to warm some of the material in order to handle it conveniently, after which it must be allowed to cool before determining the accurate weight.

The sample should now be placed in the oven, where it is allowed to remain for a period of five hours, during which time the temperature as shown by the thermometer in bitumen should not vary at any time more than  $2^{\circ}\text{C}$ . from  $163^{\circ}\text{C}$ . The sample is then removed from the oven, allowed to cool, and reweighed. From the difference between this weight and the total weight before heating, the percentage of loss on the amount of material taken is calculated.

### *New Method*

#### *Equipment:*

- 1 constant-temperature oven with revolving shelf. (Fig. 20.)
- 1 chemical thermometer reading to  $250^{\circ}\text{C}$ .
- 2 tin boxes, 5.5 centimeters in diameter by 3.5 centimeters deep.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

*Method.*—This method is substantially the “Standard Test for Loss on Heating of Oil and Asphaltic Compounds” recommended to the American Society for Testing Materials in 1916 by its committee D-4. The following description is, however, limited to a single type of apparatus and the directions are more specific than those given in the method mentioned.

A Frease electric oven with thermo-regulator and equipped with a motor and revolving shelf, as shown in Fig. 20, will be found convenient for this test. The door of the oven is provided with a glass window for observing the thermometer, which is entirely enclosed. The revolving shelf (see Fig. 21) was devised by Fitch as a means of equalizing the temperature of

a number of samples tested at the same time without reference to variations in temperature at various positions in the oven.

A circular tin box, approximately 5.5 centimeters in diameter and 3.5 centimeters deep, is first accurately weighed. The American Can Company's Gill style ointment box, deep pattern, is most commonly used for this purpose. About 50 grams of the

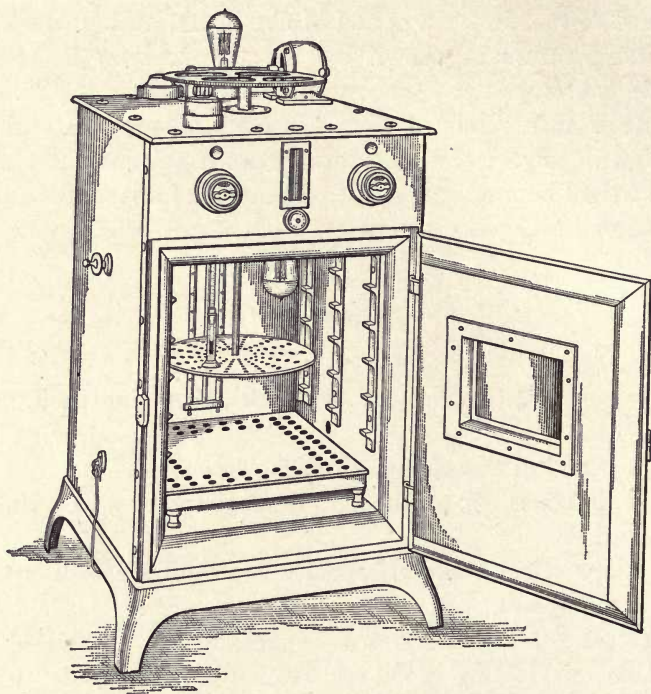


FIG. 20. Frease Electric Oven with Revolving Shelf

material under examination are then accurately weighed into the box, and this weight should not vary more than 0.2 gram from the specified amount. The box and sample are then placed in the oven, which is first brought to the prescribed temperature, usually  $163^{\circ}\text{C.}$ , and maintained to within 1 degree of that temperature throughout the test. The temperature reading is obtained from a thermometer whose bulb is immersed in a dummy sample of the material resting in the same relative position as





by volatilization will occur for different volumes or weights of a given sample which is tested. This is particularly true for materials that show a high loss under the test, and as the consistency of the residue is largely dependent upon the percentage of loss by volatilization, it follows that for a given temperature and time of test the consistency of the residue will vary with the relation of the diameter of the container and the weight of sample tested. It will be noted that in the two methods above described the diameter specified for the container is approximately the same, but that one method specifies a 20-gram sample while the other specifies a 50-gram sample. In reporting results, therefore, it is customary to indicate the weight of sample tested as well as the temperature and time of test. The 20-gram sample has been most commonly used for the harder grades of asphalt cements, but, as this amount does not afford sufficient depth for a determination of the penetration of the residue obtained from the softer asphalt cements, a strong preference is being shown for the 50-gram sample.

### *Value of the Volatilization Test*

The volatilization test at  $163^{\circ}\text{C}$ . is commonly made upon petroleum and asphalt products but seldom upon tar products. The temperature was originally selected as being that at which a refined asphalt is usually fluxed to produce an asphalt cement. With few exceptions it may also be considered as the maximum allowable temperature to heat bituminous materials for direct use. Materials which are likely to be heated to this temperature should show a low loss under the volatilization test and the residue should not show an undue amount of hardening as compared with the original sample. When testing fluid products, such as carpeting mediums, the residue from the volatilization test may be subjected to the float test if too soft for its penetration to be determined. This is particularly advisable where the float test can be made upon both the original material and its residue. In some cases the volatilization test is made at other temperatures than  $163^{\circ}\text{C}$ . Thus the ten-

dency of a material to harden very rapidly after use is shown by a test at  $100^{\circ}$ ,  $105^{\circ}$ , or  $110^{\circ}$  C. If the test is to be made upon a tar product, a relatively low temperature should be used, as  $163^{\circ}$  C. is unnecessarily severe for tars, owing to the fact that it is never necessary to heat them to this temperature during use, and if they are so heated they are apt to be seriously injured. In general, for a given type of residual product, the loss by volatilization decreases with increase in specific gravity, flash point, and specific viscosity or float test, and decrease in penetration if it is a semisolid or solid.

## DEHYDRATION—DETERMINATION OF WATER

*Equipment:*

- 1 800 cubic centimeter cylindrical copper still with ring burner and rubber tubing.
- 1 Bunsen burner.
- 2 iron stands.
- 2 small iron clamps.
- 1 iron ring support.
- 1 long piece of glass tubing, bent for a condenser.
- 1 special form separatory funnel with graduated stem.

*Method.*—When it is desired to dehydrate a bituminous material prior to making other tests or to determine the percentage of water which it contains, a cylindrical copper still with ring burner, as shown in Fig. 22, will prove convenient. From 250 to 500 cubic centimeters of the material are accurately weighed into the still and a thin paper gasket tightly clamped between the still and still head. The apparatus is then set up as shown in Fig. 22. A low flame is then applied to the upper part of the retort and the heating slowly and carefully continued until the volume of water in the special graduated form of separatory funnel shows no further increase. The volume of water collected, which settles to the bottom of the funnel, is noted and calculated upon a percentage basis of the original sample. The water is then drained from the separatory funnel and the supernatant layer of oil is run into and thoroughly

mixed with the contents of the still, which should first be cooled to below  $100^{\circ}\text{C}$ .

### *Value of Test*

Because of the tendency of bituminous materials containing only a small amount of water to foam when heated, the dehydration test is of particular value in preparing materials for

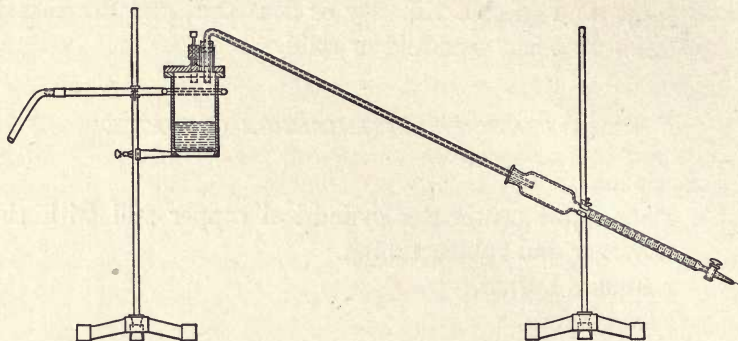


FIG. 22. Dehydrating Apparatus

any of the consistency and heat tests. It is also of service in actually determining the amount of water present which may have a marked effect upon both the chemical and physical properties of the material. When making a determination of water in semisolid and solid products, it is sometimes advisable to first render the sample fluid by the addition of benzol before attempting the distillation.

## DISTILLATION TEST

### *Flask Method*

#### *Equipment:*

- 1 250 cubic centimeter Engler distillation flask.
- 1 chemical thermometer reading from  $-5^{\circ}\text{C}$ . to  $400^{\circ}\text{C}$ .
- 1 special form condenser with rubber tubing.
- 6 25 cubic centimeter glass cylinders graduated to 0.1 cubic centimeter.



- 1 iron stand.
- 1 iron ring support 9 centimeters in diameter.
- 2 cork-lined universal clamps.
- 1 tin shield, 20 centimeters long and 9 centimeters in diameter.
- 1 pinch cock.
- 1 Bunsen burner with rubber tubing.
- 1 rough balance, capacity 1 kilogram, sensitive to 0.1 gram.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

*Method.*—This method has been recently recommended to The American Society for Testing Materials by its Committee D-4 for adoption as standard. The exact wording of the proposed standard method has not here been followed, however, as it has been thought advisable to include a few additional details for the use of beginners.

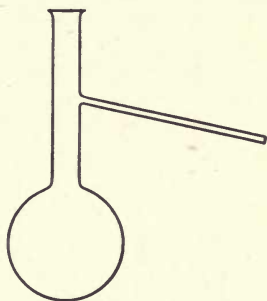


FIG. 23. Engler Distilling Flask

The Engler flask, shown in Fig. 23, should meet the following requirements within an allowance of 3 per cent variation:

Diameter of bulb.....	8.0 cm.
Length of neck.....	15.0 cm.
Diameter of neck.....	1.7 cm.
Length of tubulature.....	11.0 cm.
Diameter of tubulature.....	0.9 cm.
Angle of tubulature.....	75°

The thermometer should be made of resistance glass such as Jena 59, Jena 19, or Verra dur. It should be filled with carbon dioxide under pressure of one atmosphere at 300° C. and should be provided with an expansion bulb at the top. It should be annealed at 400° C. for 96 hours and slowly cooled. It should be graduated in single degrees Centigrade from -5° C. to 400° C., the length of the graduations from 0° to 400° C. being from 29 to 31 centimeters and the diameter of the stem from 6 to 8 millimeters. Starting at a temperature of 26° C. the

mercury should pass the  $90^{\circ}$  mark in not less than 6 seconds when plunged into a free flow of steam.

Before use in the distillation test on bituminous materials, the thermometer should be calibrated by setting up the entire apparatus, as shown in Fig. 24, and noting the apparent temperatures at which three chemically pure substances of known boiling point distill. One hundred cubic centimeters of distilled

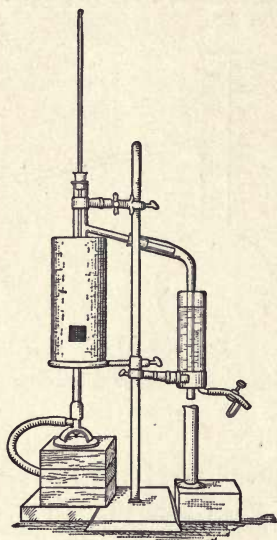


FIG. 24. Distillation Apparatus—Flask Method

water, chemically pure naphthalene and chemically pure benzophenone are used for this purpose. Their normal boiling points are  $100^{\circ}$ ,  $218.2^{\circ}$ , and  $305^{\circ}$  C. respectively. The boiling points of these substances, of course, vary with the barometric pressure, but if the thermometer is calibrated at a time when the barometer indicates about the average pressure for a given laboratory, the variations in results due to varying pressures when the thermometer is afterwards used for distillation will be no greater than the probable errors in distillation. The intermediate readings of the thermometer are obtained by plotting the results as shown in Fig. 25.

Thus, if it is desired to make a cut at  $170^{\circ}$  C. during a distillation, it is evident that the cut should be made under with this particular thermometer at an observed temperature of  $167^{\circ}$ . The correctness of the thermometer should be checked at two temperatures after each third distillation until the thermometer is thoroughly seasoned.

The condenser used in connection with the distilling flask may be made by bending a standard condenser tube as shown in Fig. 24 and passing it through a tight-fitting stopper placed in the bottom of an ordinary Welsbach lamp chimney. A bent tube carrying a short length of rubber tubing and a pinch cock is also fitted into the stopper for the purpose of draining the

condenser of cooling water if desired. The condenser tube when straight should have the following dimensions:

Adapter.....	70 mm.
Length of straight tube.....	185 mm.
Width of tube.....	12-15 mm.
Width of adapter end of tube.....	20-25 mm.

If the material to be tested contains water, it should first be dehydrated or otherwise it is apt to foam over during

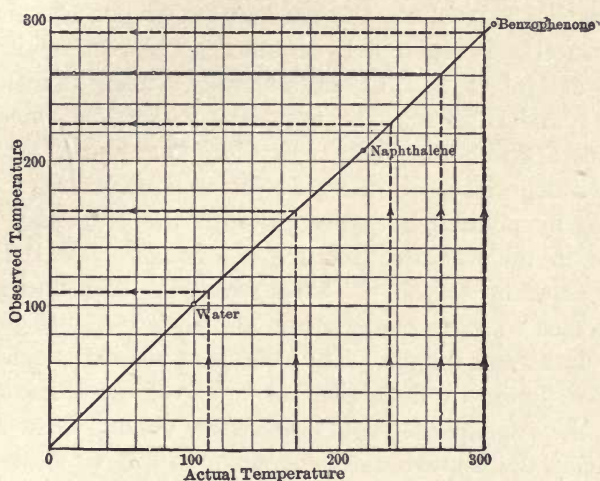


FIG. 25. Example of Thermometer Calibration Chart

distillation. The specific gravity of the water-free material is determined so that the weight of 100 cubic centimeters may be calculated. In making the test the flask should first be supported in a vertical position on one pan of the rough balance and its tare accurately obtained. After warming, if necessary, and thoroughly stirring the sample, 100 cubic centimeters are poured into the tared flask and weighed. A cork stopper carrying the thermometer is then inserted in the neck of the flask so that the top of the bulb is opposite the middle of the tubulature. The entire apparatus is then set up as shown in Fig. 24. A tin shield with small sight hole for observing the Bunsen flame surrounds the flask and burner in order to obviate the influence of drafts. The glass graduates which have previously



been weighed to within 0.1 gram are used as receivers for the distillate fractions. In preparing for the test it will be found convenient to mark permanently on the foot of each graduate its weight.

The material should be heated gradually by means of the Bunsen burner, and the heat should be so regulated as to maintain distillation at the constant rate of 1 cubic centimeter per minute. When the thermometer just passes a temperature corresponding to 110° C., the graduated cylinder containing the first fraction is replaced by another. The receiver is changed again at 170° C., 235° C., and at 270° C., using as many graduated cylinders as may be necessary without allowing any to become filled above the 25 cubic centimeter mark. When solid matter deposits upon the sides of the condenser, it may be melted by pouring hot water through the condenser, and collected in the fraction to which it belongs. The last fraction is collected up to 300° C., after which the flask and graduates are cooled to room temperature, and their contents determined by volume and weight. The volume of pitch remaining in the retort is found by deducting the total volume of the distillates from the original 100 cubic centimeters taken. Note should be made of the approximate volume of solids which precipitate from the distillates upon cooling to 25° C.

The results obtained are calculated in percentages by volumes and weights to tenths of 1 per cent and reported as follows:

Distillate	Per cent by volume	Per cent by weight
1. Water or ammoniacal liquor . . . . .	.....	.....
2. First light oils to 110° C. . . . .	.....	.....
3. Second light oils 110° C. to 170° C. . . . .	.....	.....
4. Heavy oils 170° C. to 270° C. . . . .	.....	.....
5. Heavy oils 270° C. to 300° C. . . . .	.....	.....
6. Pitch residue . . . . .	.....	.....

### *Retort Method*

#### *Equipment:*

1 8 oz. stoppered glass retort.

1 chemical thermometer reading from -5° C. to 400° C.

- 1 condensing tube.
- 6 25 cubic centimeter glass cylinders or bottles.
- 2 iron stands.
- 2 cork-lined universal clamps.
- 1 iron ring support.
- 1 tin shield.
- 1 Bunsen burner with rubber tubing.
- 1 rough balance, capacity 1 kilogram, sensitive to 0.1 gram.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

*Method.*—While the flask method is well adapted to the distillation of all water-free bituminous materials and has been more accurately standardized, the following method has been

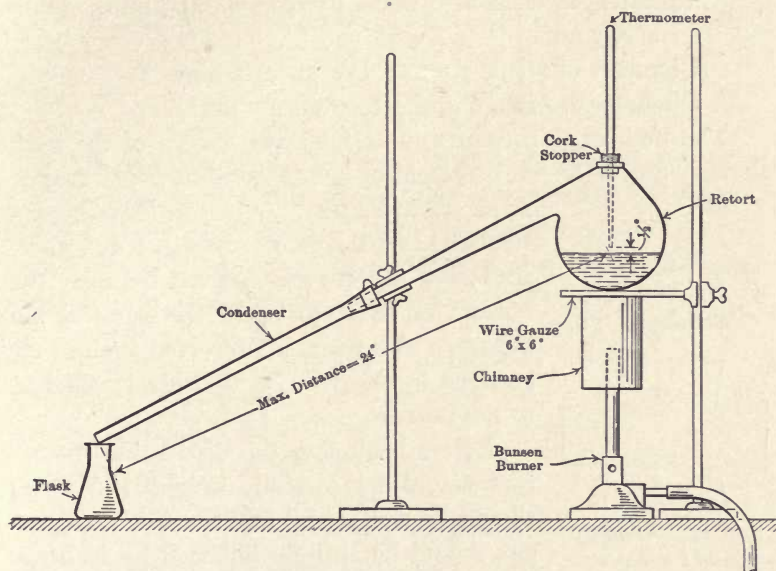


FIG. 26. Distillation Apparatus.—Retort Method

and is now widely used in the distillation of creosoting oils. It is known as the Standard Creosoters' Method. The following description is essentially the same as published in Bulletin 65 of the American Engineering, Railway, and Maintenance of Way Association, although the wording is different to some

extent, and certain details have been added in accordance with the tentative method recommended in 1915 by Committee D-7 of the American Society for Testing Materials.

The retort, as shown in Fig. 26, should have a capacity of eight ounces up to the bend of the neck when the bottom of the retort and the mouth of the offtake are in the same horizontal plane. The thermometer should be nitrogen-filled and divided into full degrees Centigrade. It should have the following dimensions:

Total length, 375 mm.; tolerance, 10 mm.

Bulb length, 14 mm.; tolerance, 1 mm.

Distance from zero mark to bottom of bulb, 30 mm.; tolerance, 4 mm.

Scale length from zero mark to  $400^{\circ}$  C., 295 mm.; tolerance, 5 mm.

Diameter of stem, 7 mm.; tolerance, 1 mm.

Diameter of bulb, 6 mm.; tolerance, 1 mm.

The bulb of the retort and at least two inches of the neck should be covered with a shield of heavy asbestos paper shaped as shown in Fig. 27. When making the distillation, two sheets of 20-mesh wire gauze, six inches square, are inserted between the bottom of the retort and the flame of the burner. The flame is protected against air currents by means of a tin shield attached to the burner.

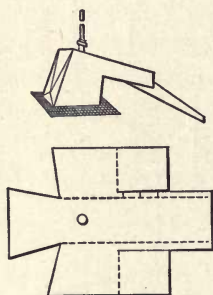


FIG. 27. Asbestos Shield for Retort

Before beginning the distillation the retort should be carefully weighed. The oil should be well mixed and if solids are present should be entirely liquefied by heating. If it contains water it should be dehydrated prior to the test. Exactly 100 grams of the water-free oil should then be weighed into the retort. The thermometer should next be inserted through a tight-fitting cork stopper in the tubulature of the retort, so that the lower end of the bulb is one-half inch from the surface of the oil. The condensing tube is then attached to the retort by a tight cork



joint and the entire apparatus set up as shown in Fig. 26. The distance between the bulb and the end of the condensing tube should be not less than 20 nor more than 24 inches.

Distillation should be conducted at the rate of from 1 to 2 drops per second and the distillate collected in weighed receivers. The condenser tube should be warmed when necessary to prevent accumulation of solid distillates. The receivers should be changed as the mercury passes the fractionating point. Fractions are collected and weighed as follows:

Up to 170° Centigrade			
From 170° to 200° Centigrade			
"	200°	"	210°
"	210°	"	235°
"	235°	"	270°
"	270°	"	315°
"	315°	"	355°

The last receiver should be removed at 355° C. without draining the condenser. The various fractions should be reported upon a percentage weight basis. In reporting results under ordinary conditions, certain of the fractions are taken collectively and the total number of fractions reduced as follows:

Up to 200° Centigrade			
200° to 210° Centigrade			
210°	"	235°	"
235°	"	315°	"
Above	315°		"

#### *Value of Distillation Test*

The distillation test as applied to tars is valuable both as a means of ascertaining their method of preparation and suitability for a given purpose. In this connection it takes the place of the volatilization test which is usually restricted to the examination of petroleum and asphalt products. All crude tars contain water, and the appearance of water in the distillate

therefore indicates a crude material, especially if accompanied by an appreciable quantity of light oil. Heavy refined tars which have become contaminated with water show a sudden rise in distillation temperature after the water has been removed. The presence of high percentages of naphthalene, anthracene, etc., is indicated by the proportion of the various distillates which solidify upon cooling. In this connection it is always well to note the character of each fraction obtained. The extent of distillation in the manufacture of the original material is indicated by the temperature at which distillation commences in the test and the relative quantities of the fractions obtained. Cut-back products usually show an abnormal amount of some given fraction as compared to the amount that would be obtained from a straight distilled residue of the same consistency. After the distillation test is completed it is often of interest to determine the melting point of the residue which, if obtained by distillation by the flask method to  $300^{\circ}\text{C.}$ , should seldom exceed  $75^{\circ}\text{C.}$  Various fractions are also frequently examined for the purpose of identifying the material.

In general, for a given type of residual material the percentage of total distillate to any given temperature decreases as the specific gravity and viscosity or float test increase and as the penetration decreases.

## SOLUBILITY TESTS ON OTHER THAN BITUMINOUS AGGREGATES

### TOTAL BITUMEN

#### *Rapid Method*

##### *Equipment:*

- 1 100 cubic centimeter Erlenmeyer flask.
- 1 500 cubic centimeter flask with side neck for filtering under pressure.
- 1 rubber stopper with one hole.

- 1 filter tube, 3.9 centimeters inside diameter.
- 1 platimun or porcelain Gooch crucible.
- 1 piece of seamless rubber tubing, about 3 centimeters in diameter and 3 centimeters long.
- 50 grams of long-fiber amphibole asbestos.
- 2 wash bottles: 1 for solvent, 1 for water.
- 1 Bunsen burner.
- 1 nichrome triangle.
- 1 iron tripod.
- 1 drying oven.
- 1 desiccator with calcium chloride.
- 1 thermometer reading from  $-10^{\circ}$  C. to  $110^{\circ}$  C.
- 1 vacuum pump and connections.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

*Method.*—As by definition all bitumen is soluble in carbon disulphide, the per cent of total bitumen in a bituminous material is ascertained by determining its solubility in carbon disulphide. This solvent is an ethereal, mobile liquid which is extremely inflammable and should therefore be handled with great care. It is very volatile and when mixed with air its vapors spontaneously ignite at a temperature slightly higher than that of live steam. It should therefore never be used in the vicinity of a free flame or even an electric hot plate. In the laboratory where carbon disulphide is used it is always advisable to have close at hand a chemical fire extinguisher or large bottle of carbon tetrachloride, which in itself is an effective fire extinguisher.

The most widely used method of making the total bitumen determination consists in dissolving the bitumen in carbon disulphide and recovering any insoluble matter by filtering the solution through an asbestos felt. The form of Gooch crucible best adapted for the determination is 4.4 centimeters wide at the top, tapering to 3.6 centimeters at the bottom, and is 2.5 centimeters deep.

For preparing the felt the necessary apparatus is arranged as shown in Fig. 28, in which *a* is the filtering flask, *b* a rubber stopper, *c* the filter tube, and *d* a section of rubber tubing which



tightly clasps the Gooch crucible, *e*. The asbestos is cut with scissors into pieces not exceeding 1 centimeter in length, after which it is shaken up with just sufficient water to pour easily. The crucible is filled with the suspended asbestos, which is allowed to settle for a few moments. A light suction is then applied to draw off all the water and leave a firm mat of asbestos in the crucible. More of the suspended material is added, and the operation is repeated until the felt is so dense that it

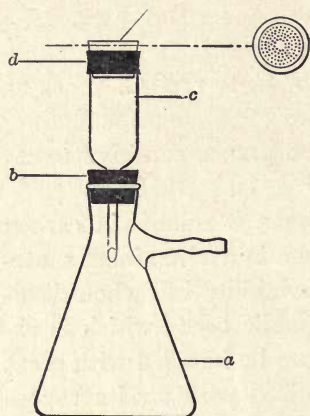


FIG. 28. Apparatus for Total Bitumen Determination

scarcely transmits light when held so that the bottom of the crucible is between the eye and the source of light. The felt should then be washed several times with water, and drawn firmly against the bottom of the crucible by an increased suction. The crucible is removed to a drying oven for a few minutes, after which it is ignited at red heat over a Bunsen burner, cooled in a desiccator and weighed.

From 1 to 2 grams of the material under examination is now placed in the Erlenmeyer flask, which has been previously weighed, and the accurate weight of the sample is obtained. One hundred cubic centimeters of chemically pure carbon disulphide are poured into the flask in small portions, with continual agitation, until all lumps disappear and nothing adheres to the bottom. The flask is then corked and set aside for 15 minutes.

After being weighed, the Gooch crucible containing the felt is set up over the dry-pressure flask, as shown in Fig. 28, and the solution of bitumen in carbon disulphide is decanted through the felt without suction by gradually tilting the flask, with care not to stir up any precipitate that may have settled out. At the first sign of any sediment coming out, the decantation is stopped and the filter allowed to drain. A small amount of carbon disulphide is then washed down the sides of the flask,

after which the precipitate is brought upon the felt and the flask scrubbed, if necessary, with a feather or "policeman," to remove all adhering material. The contents of the crucible are washed with carbon disulphide until the washings run colorless. Suction is then applied until there is practically no odor of carbon disulphide in the crucible, after which the outside of the crucible is cleaned with a cloth moistened with a small amount of the solvent. The crucible and contents are dried in the hot-air oven at  $100^{\circ}$  C. for about 20 minutes, cooled in a desiccator, and weighed. If any appreciable amount of insoluble matter adheres to the flask, it should also be dried and weighed, and any increase over the original weight of the flask should be added to the weight of insoluble matter in the crucible. The total weight of insoluble material may include both organic and mineral matter. The former, if present, is burned off by ignition at a red heat until no incandescent particles remain, thus leaving the mineral matter or ash, which can be weighed by cooling. The difference between the total weight of material insoluble in carbon disulphide and the weight of substance taken equals the total bitumen, and the percentage weights are calculated and reported as total bitumen, and organic and inorganic matter insoluble, on the basis of the weight of material taken for analysis.

Results are usually reported as follows:

Total Bitumen (or Material Soluble in $\text{CS}_2$ ).....	.....%
Organic Matter Insoluble (or Free Carbon in the Case of Tars) ..	.....%
Inorganic Matter Insoluble (or Ash).....	.....%
	<hr/>
	100.00%

This method is quite satisfactory for straight oil and tar products, but where certain natural asphalts are present it will be found practically impossible to retain all of the finely divided mineral matter on an asbestos felt. It is, therefore, generally more accurate to obtain the result for total mineral matter by direct ignition of a 1-gram sample in a platinum crucible or to use the result for ash obtained in the fixed carbon test. The total bitumen is then determined by deducting from 100 per cent the sum of the percentages of total mineral matter and



organic matter insoluble. If the presence of a carbonate mineral is suspected, the percentage of mineral matter may be most accurately obtained by treating the ash from the fixed carbon determination with a few drops of ammonium carbonate solution, drying at  $100^{\circ}\text{C}$ ., then heating for a few minutes at a dull-red heat, cooling, and again weighing.

When difficulty in filtering is experienced—for instance, when Trinidad asphalt is present in any amount—a period of longer subsidence than 15 minutes is necessary and the following method may then be used:

### *Long Method*

*Equipment.*—Same as for the rapid method except that two 150 cubic centimeter Erlenmeyer flasks are required in place of one 100 cubic centimeter flask.

*Method.*—The following method was adopted in 1911 by The American Society for Testing Materials and is particularly adapted for use in connection with products such as Trinidad asphalt which contain a relatively high percentage of very finely divided mineral matter that either passes through or tends to clog the filtering medium. While it may also be used in connection with other products, it will in some cases be found to give somewhat lower results in the percentage of total bitumen, owing to the tendency of some organic matter which was originally soluble to precipitate in a comparatively dilute solution of carbon disulphide upon prolonged standing. The Committee in presenting this method stated that they wished it understood that they do not recommend it as the best for general use, as it is longer and in many cases gives no better results than other more expeditious methods, but only as a method to be resorted to in case of dispute, as it seems to have the widest range of applicability of any of the methods which they had considered.

"After drying, from 2 to 15 grams (as may be necessary to insure the presence of 1 to 2 grams of pure bitumen) are weighed into a 150-c.c. tared Erlenmeyer flask and treated with 100 c.c. of carbon disulphide. The flask is then loosely corked and shaken from time to time until all large particles of the material have been broken up. It is then set aside for 48 hours to settle. The solution is decanted into a similar flask that has been previously weighed. As much of the solvent is poured off as possible without disturbing the residue. The contents of the first flask are again treated with fresh carbon disulphide, shaken as before, and then put away with the second flask for 48 hours to settle.

"The liquid in the second flask is then carefully decanted upon a weighed Gooch crucible, 3.2 cm. in diameter at the bottom, fitted with an asbestos filter, and the contents of the first flask are similarly treated. The asbestos filter is made of ignited long-fiber amphibole, packed in the bottom of a Gooch crucible to the depth of not over  $\frac{1}{8}$  in. In filtering, no vacuum is to be used and the temperature is to be kept between  $20^{\circ}$  and  $25^{\circ}\text{C}$ . After passing the liquid contents of both flasks through the filter, the residue on the filter is thoroughly washed and the residues remaining in them are shaken with more fresh carbon disulphide and allowed to settle for 24 hours, or until it is seen that a good subsidence has taken place. The solvent in both flasks is then again decanted through the filter and the residues remaining in them are washed until the washings are practically colorless. All washings are to be passed through the Gooch crucible.



"The crucible and both flasks are then dried at 125° C. and weighed. The filtrate containing the bitumen is evaporated, the bituminous residue burned, and the weight of the ash thus obtained added to that of the residue in the two flasks and the crucible. The sum of these weights deducted from the weight of substance taken gives the weight of soluble bitumen."

### *Value of Total Bitumen Determination*

The methods above described are best suited for the determination of total bitumen in comparatively pure bituminous materials, although they are also applicable to the examination of fine bituminous aggregates when it is not desired to recover the mineral matter for the purpose of grading it or making a mechanical analysis. As, however, it is usually desired to grade the aggregate, one of the methods described under "Extraction of Bituminous Aggregates and Recovery of Bitumen" is usually to be preferred for such products.

As bituminous materials are sometimes purchased upon their per cent of total bitumen, the determination may be necessary as a basis of payment. All bituminous distillates produced by fractional distillation are completely soluble in carbon disulphide. Residual petroleum products should also be almost completely soluble. A small amount of still ash and extraneous organic matter is, however, usually present, and an allowance of approximately 0.5 per cent may therefore be made for total material insoluble in carbon disulphide. If the organic matter insoluble exceeds 0.5 per cent, there is strong indication that the petroleum has been cracked or locally overheated with possible resultant injury to the finished product. Native asphalts and products produced by fluxing the native asphalts ordinarily contain appreciable amounts of both mineral matter and organic matter insoluble in carbon disulphide. Gilsonite is one of the few exceptions to the rule. Where the exact percentage of total insoluble matter is known for a refined native asphalt, it is possible to compute with considerable accuracy the amount of such asphalt present in an asphalt cement produced by fluxing it. In such computation the petroleum flux is assumed to be completely soluble unless information to the contrary has been secured. Practically all of the material in tar prod-

ucts which is insoluble in carbon disulphide consists of organic matter or free carbon, and the percentage of free carbon is thus determined. Refined water-gas tars may be distinguished from refined coal tars of the same consistency by their relatively small percentage of free carbon. Gas-house coal tars, particularly those produced from horizontal retorts, may usually be distinguished from coke-oven tars and vertical-retort tars by their relatively high percentage of free carbon. For refined tars of the same viscosity or float test the specific gravity becomes higher as the solubility in carbon disulphide decreases. The same is true of asphalt cements produced from the same native asphalt if such asphalt contains insoluble material. As the solubility of a bituminous material in carbon disulphide is entirely dependent upon the non-bituminous constituents present, it is evident that no definite relations can be expressed between the physical and chemical properties of the actual bitumen present and the solubility of the material in this solvent. In other words, irrespective of specific gravity, consistency and other properties, all pure bitumen is dissolved by carbon disulphide. An oily, greasy distillate is therefore just as much bitumen as a hard, brittle residue.

#### ASPHALTENES OR BITUMEN INSOLUBLE IN PARAFFIN NAPHTHA

*Equipment.*—Same as for the total bitumen determination, rapid method.

*Method.*—This determination is made in the same general manner as the rapid method for total bitumen, except that 100 cubic centimeters of 86° to 88° B. paraffin naphtha, at least 85 per cent by volume distilling between 35° C. and 65° C., is employed as a solvent instead of carbon disulphide.

Petroleum naphthas are not definite chemical compounds, but are composed of a number of hydrocarbons which vary in character and quantity according to the petroleum from which they have been distilled. Their solvent action upon petroleum and asphalt products therefore varies greatly. Thus naphthas produced from asphaltic petroleums, consisting mainly

of naphthene and polymethylene hydrocarbons, are much more powerful solvents of the heavier asphaltic hydrocarbons than are the paraffin naphthas. Moreover, the solvent power of any given type of naphtha increases with its specific gravity. As the main object of the naphtha insoluble bitumen test is to separate the heavier hydrocarbons of an asphaltic nature from the other constituents, a light paraffin distillate is usually employed. One of 86° B. gravity, distilling between 40° and 65° C., has been used to a considerable extent, as is also an 88° B. naphtha, the solvent power of which is approximately the same as that of the 86° B. products. Heavier naphthas, such as 66° B. and 72° B., are also less frequently used.

As the percentage of asphaltenes as determined by test is largely dependent upon the gravity and type of naphtha used as a solvent, a report of results should state the gravity of the naphtha and preferably its boiling-point limits as well. The determination is not entirely satisfactory, and investigations have been conducted with a view to finding some solvent of definite chemical composition which will satisfactorily replace naphtha and more nearly insure uniform results on the part of different analysts. Ethyl alcohol and ether have been suggested for this purpose, but have not been generally used.

Considerable difficulty is sometimes experienced in breaking up some of the heavy semisolid bitumens; the surface of the material is attacked, but it is necessary to remove some of the insoluble matter in order to expose fresh material to the action of the solvent. It is therefore advisable to heat the sample after it is weighed, allowing it to cool in a thin layer around the lower part of the flask. If difficulty is still experienced in dissolving the material, a rounded glass rod will be found convenient for breaking up the undissolved particles. Not more than one-half of the total amount of naphtha required should be used until the sample is entirely broken up. The balance of the 100 cubic centimeters is then added, and the flask is twirled a moment in order to mix the contents thoroughly, after which it is corked and set aside for 30 minutes.

In making the filtration the utmost care should be exercised



to avoid stirring up any of the precipitate, in order that the filter may not be clogged and that the first decantation may be as complete as possible. The sides of the flask should then be quickly washed down with naphtha and, when the crucible has drained, the bulk of insoluble matter is brought upon the felt. Suction may be applied when the filtration by gravity almost ceases, but should be used sparingly, as it tends to clog the filter by packing the precipitate too tightly. The material on the felt should never be allowed to run entirely dry until the washing is completed, as shown by the colorless filtrate. When considerable insoluble matter adheres to the flask no attempt should be made to remove it completely. In such cases the adhering material is merely washed until free from soluble matter, and the flask is dried with the crucible at  $100^{\circ}$  C. for about one hour, after which it is cooled and weighed. The percentage of bitumen insoluble is reported upon the basis of total bitumen taken as 100.

The difference between the material insoluble in carbon disulphide and in the naphtha is the bitumen insoluble in the latter. Thus, if in a certain instance it is found that the material insoluble in carbon disulphide amounts to 1 per cent and that 10.9 per cent is insoluble in naphtha, the percentage of bitumen insoluble would be calculated as follows:

$$\frac{\text{Bitumen insoluble in naphtha}}{\text{Total bitumen}} = \frac{10.9 - 1}{100 - 1} = 10 \text{ per cent.}$$

### *Value of the Determination*

While the class of bodies known as asphaltenes in petroleum and asphalt products are of variable nature, it may be said that in general they tend to give body and consistency as well as cementitiousness to the materials in which they occur. Thus the native asphalts carry a high percentage of asphaltenes while the fluid native bitumens or petroleums carry a relatively low percentage. Paraffin petroleums contain less than asphaltic petroleums, and petroleum distillates, which possess no cementitiousness, contain none at all. Upon distillation of a petro-

leum the asphaltenes tend to concentrate in the residuum and under certain conditions new asphaltenes are actually formed. The blowing process, by causing what is known as nucleus condensation of certain of the lighter hydrocarbons, forms asphaltenes, which are directly responsible for the gradual change of the fluid to a semisolid or solid. Irrespective of their method of manufacture, asphalt cements of a given penetration at 25° C. usually carry asphaltenes within comparatively narrow percentage limits and, at least for a given type of asphalt cement, the penetration is found to decrease as the percentage of asphaltenes increases. The percentage of asphaltenes is usually additive and, for mixtures, may be calculated with reasonable accuracy from the amounts present in the individual constituents of the mixture. In fluxing a hard asphalt to any higher penetration, the amount of flux that will be required will, therefore, depend to a considerable extent upon the percentage of asphaltenes present in the flux. The higher this percentage the more will be required. Owing to the unavoidable variations to which this determination is subject, specification limitations of the per cent of bitumen insoluble in naphtha are necessarily wider than though a definite chemical compound was the solvent used. The determination is useful as a means of identification in some instances, particularly when considered in connection with other tests. Of the solid native bituminous materials, Gilsonite shows a high percentage of asphaltenes, usually over 45 per cent, while the bitumen of grahamite appears to be almost entirely composed of asphaltenes. While the test is not generally applied to tars, it is of interest to note that the heavy refined tars are almost entirely insoluble in light paraffin naphthas.

#### CARBENES OR BITUMEN INSOLUBLE IN CARBON TETRACHLORIDE

*Equipment.*—Same as for the total bitumen determination, rapid method.

*Method.*—This determination is made in exactly the same manner as the total bitumen determination, except that chemically pure carbon tetrachloride is used as a solvent in place of carbon disulphide.

Carbon tetrachloride is a volatile, non-inflammable liquid, which is almost as powerful a solvent for bitumen as carbon disulphide. It is widely used as a fire extinguisher, and it is advisable to keep a bottle of the material in the laboratory for that purpose. It is a definite chemical compound having the formula  $\text{CCl}_4$ . In the presence of some organic matter, especially in direct sunlight, it tends to decompose slowly and liberate hydrochloric acid, although under ordinary conditions it is chemically stable. With the bitumen which it first dissolves it may later react to precipitate a small amount of insoluble material. This property must be taken into account in its use as a solvent and a short period of digestion is therefore adopted in order to prevent a determination of more insoluble material than is really present.

The percentage of bitumen insoluble in carbon tetrachloride is reported upon the basis of total bitumen taken as 100. The difference between the material insoluble in carbon disulphide and in carbon tetrachloride is the bitumen insoluble in the latter. The method of calculating results to be reported is illustrated by the following example, if the amount of material insoluble in  $\text{CS}_2$  is 0.1% and the amount insoluble in  $\text{CCl}_4$  is 0.3%.

$$\frac{\text{Bitumen insoluble in } \text{CCl}_4}{\text{Total bitumen}} = \frac{0.3 - 0.1}{100 - 0.1} = \frac{0.2}{99.9} = 0.2\%$$

### *Value of the Determination*

The presence of carbenes in petroleum and asphalt products is indicative of unnecessarily high temperatures in their production. Most carefully prepared petroleum residuums are as completely soluble in carbon tetrachloride as in carbon disulphide. Incipient cracking due to local overheating or prolonged exposure to high temperatures is indicated by the presence of carbenes, although no coke or organic matter insoluble in carbon disulphide may have been formed. Where carbenes in petroleum residuums are accompanied by an appreciable amount of organic matter insoluble in carbon disulphide, indications point strongly to injury of the material by decomposition of some of the hydro-



carbons present. Some native asphalts, such as the Trinidad and Cuban products, are slightly more soluble in carbon tetrachloride than in carbon disulphide, while others that have been produced in nature at relatively high temperatures show appreciable quantities of carbenes. Grahamite in particular may show as high as 60 or 70 per cent of carbenes. With the exception of grahamite, however, the percentage of carbenes in most petroleum and asphalt products for use in highway engineering is commonly specified as less than 0.5%. There is no direct relation which may be expressed between the percentage of carbenes and other physical and chemical properties of a material. Thus a fluid petroleum residuum carelessly manufactured may contain a relatively large amount, while a solid residuum carefully prepared from the same petroleum may be entirely soluble in carbon tetrachloride. All distillates produced by the fractional distillation of bituminous materials are completely soluble in this solvent. As a rule, tars are not as soluble in carbon tetrachloride as in carbon disulphide, but the difference in solubility appears to bear no relation to the amount of free carbon which is present in the tar. In the routine examination of tars, carbon tetrachloride is not employed as a solvent, and the term "carbenes" is therefore limited to constituents of petroleum and asphalt products.

#### DIMETHYL SULPHATE TEST

*Equipment.*—Same as for the distillation test, flask method, and in addition 3 10 cubic centimeter glass cylinders, with ground-glass stoppers, graduated to 0.2 cubic centimeters.

*Method.*—The dimethyl sulphate test is used to detect the presence of petroleum or asphalt products in tars and is usually made upon high boiling distillates obtained from a sample of the material which has been subjected to the distillation test, flask method. For this purpose the pitch residue which has been obtained from the distillation test, after cooling and weighing, is again heated in the original Engler flask and fractions are taken at 350° C. and 370° C. These fractions, together with

the 270–300° C. fraction previously obtained, are separately stirred, and, if necessary, heated to dissolve solids which may be present.

Four cubic centimeters of distillate from each fraction are separately shaken with 6 cubic centimeters of dimethyl sulphate in a 10 cubic centimeter cylinder. After standing 30 minutes any resulting supernatant layer of insoluble oil is read and calculated to its percentage by volume of the sample of distillate taken. The results are reported as follows:

Fractions	Per cent distillate	Per cent of distillate insoluble in dimethyl sulphate
270° to 300° C. ....	.....	.....
300° to 350° C. ....	.....	.....
350° to 375° C. ....	.....	.....

Great care should be exercised in handling the dimethyl sulphate as it is very poisonous, both the liquid and its vapors being extremely irritating to the skin.

### *Value of the Test*

Petroleum and asphalt distillates are insoluble in dimethyl sulphate while tar distillates are completely soluble. Mixed distillates are therefore only partially soluble. Sufficiently high boiling distillates are tested to insure the presence of fractions of the petroleum or asphalt product if either is present. The test is mainly qualitative, but is valuable when as little as 3 per cent of petroleum or asphalt products are present in the tar.

## MISCELLANEOUS TESTS

### FIXED CARBON AND ASH

#### *Equipment:*

- 1 iron ring support (ring 7.5 cm. in diameter).
- 1 platinum or nichrome triangle.
- 1 Bunsen burner and rubber tubing.

- 1 platinum crucible with a tight-fitting cover (weight complete, from 20 to 30 grams).
- 1 crucible tongs.
- 1 desiccator with calcium chloride.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

*Method.*—This is a purely arbitrary test for bituminous materials which has been borrowed from the ordinary methods for coal analysis. Described here somewhat more in detail, it is the same as that adopted by The American Society for Testing Materials in 1911. The determination is made in accordance with the method described for coal in the Journal of the American Chemical Society, 1899, Volume 21, page 1116. One gram of the material is placed in a platinum crucible weighing from 20 to 30 grams and having a tightly fitting cover. It is then heated for seven minutes over the full flame of a Bunsen burner, as shown in Fig. 29. The crucible should be supported on a platinum or nichrome triangle with the bottom from 6 to 8 centimeters above the top of the burner. The flame should be fully 20 centimeters high when burning freely, and the determination should be made in a place free from drafts. The upper surface of the cover should burn clear, but the under surface should remain covered with carbon, excepting in the case of some of the more fluid bitumens, when the under surface of the cover may be quite clean.

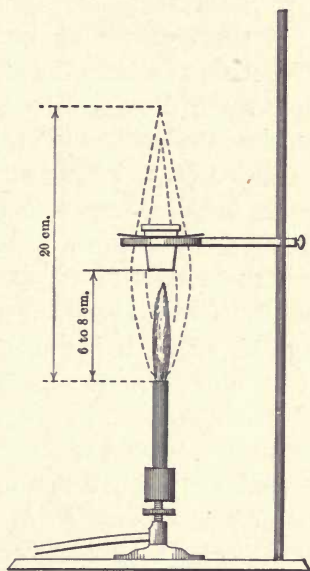


FIG. 29. Apparatus for Determination of Fixed Carbon

The crucible is removed to a desiccator and when cool is weighed, after which the cover is removed, and the crucible is placed in an inclined position over the Bunsen burner and



ignited until nothing but ash remains. Any carbon deposited on the cover is also burned off. The weight of the ash remaining is deducted from the weight of the residue after the first ignition of the sample. This gives the weight of the so-called fixed or residual carbon, which is calculated on a basis of the total weight of the sample, exclusive of mineral matter. Thus, if a one-gram sample yields upon the first heating 0.13 grams residue and after final ignition 0.03 grams of ash, the percentage of fixed carbon is calculated as follows:

$$\frac{0.13 - 0.03}{1.00 - 0.03} = \frac{0.10}{0.97} = 10.72\%$$

If the presence of a carbonate mineral is suspected, the percentage of mineral matter may be most accurately obtained by treating the ash with a few drops of ammonium carbonate solution, drying at 100° C., then heating for a few minutes at a dull-red heat, cooling and weighing.

An excellent form of crucible for this test is shown in Fig. 29. It has a cover with a flange 4 millimeters wide, fitting tightly over the outside of the crucible, and weighs complete about 25 grams. Owing to sudden expansion in burning some of the more fluid bitumens, it is well to hold the cover down with the end of the tongs until the most volatile products have burned off.

Some products, particularly those derived from Mexican petroleum, show a tendency to suddenly expand and foam over the sides of the crucible in making this determination, and no method of obviating this trouble without vitiating the result has thus far been forthcoming. Recent experiments in the laboratory of the Office of Public Roads and Rural Engineering indicate that the difficulty may in many cases be overcome by placing a small piece of platinum gauze over the sample and about midway of the crucible. The gauze should be so cut or bent as to touch the sides of the crucible at all points, and is of course weighed in place in the crucible before and after ignition.

*Value of the Test*

To be of any real value the fixed carbon determination should be made exactly as described in the preceding paragraphs. It is not an exceedingly accurate test, and any attempt at variation in the method of heating or other conditions specified will often greatly affect the results obtained. When properly made, the fixed carbon test may serve as a means of identifying the type of petroleum from which a fluid or semisolid residuum has been manufactured. Paraffin petroleums and all petroleum distillates yield little or no fixed carbon. Asphaltic petroleums and their residual products always yield fixed carbon, but in varying amounts, according to their origin or method of manufacture. For a given viscosity, float test, or penetration, blown petroleums show a very low percentage of fixed carbon and straight distilled Mexican petroleum residuums a relatively high percentage. For any type of petroleum residuum, fixed carbon increases as distillation progresses. It is therefore accompanied by increase in specific gravity, percentage of asphaltenes, viscosity and float test, and decrease in penetration. Native asphalts usually yield from 11 to 15 per cent of fixed carbon, although the harder varieties sometimes run considerably higher, and in the case of grahamite may reach 50 per cent or over. The test is seldom applied to tars, or tar products, owing to the presence of free carbon which interferes with the determination. If made upon tars, the weight of free carbon in the one-gram sample should be determined by test and deducted from the weight of residue from the first heating before calculating the percentage of fixed carbon.

In interpreting the fixed carbon determination it should be remembered that the fixed carbon is actually produced from the material by destructive distillation and does not exist as such in the original material. It is therefore quite distinct from free carbon, which, if determined to exist at all, is actually present in the original material and is merely separated from the other constituents by means of a solvent.

The amount and character of the ash often serves as an aid to

identification of the material. Thus petroleum products seldom yield more than 0.2 and usually less than 0.1 per cent ash, while the native asphalt products yield considerably more, in certain cases running over 30 per cent. Moreover, the ash of many of the native asphalts shows a characteristic color and texture.

#### PARAFFIN SCALE DETERMINATION

##### *Equipment:*

- 1 one-half pint iron retort. (Fig. 30.)
- 1 piece iron tubing, 30 inches long.
- 2 100 cubic centimeter Erlenmeyer flasks.
- 1 500 cubic centimeter (16-oz.) flask, with side neck for filtering under pressure.
- 1 freezing apparatus. (Fig. 31.)
- 1 6-inch test tube,  $\frac{3}{4}$ -inch diameter.
- 1 analytical balance, capacity 100 grams, sensitive to 0.1 milligram.
- 1 rough balance, capacity 1 kilogram, sensitive to 0.1 gram.
- 1 wash bottle.
- 1 pint tin cup, seamless.
- 1 vacuum pump and connections.
- 1 glass crystallizing dish, 50 millimeters in diameter.
- 1 steam bath.
- 1 desiccator with calcium chloride.
- 1 4-inch steel spatula.
- 1 Bunsen burner with rubber tubing.
- 2 iron stands with retort clamp, and 1 ring.

*Method.*—Fifty grams of the material under examination should be weighed into the tared iron retort as shown in Fig. 30, and distilled as rapidly as possible to dry coke. The distillation should be complete in not over 25 minutes. The distillate is caught in a 100 cubic centimeter Erlenmeyer flask, the weight of which has been previously ascertained. During the early stages of distillation a cold, damp towel wrapped around the stem of the retort will serve to condense the distillate. After high temperatures have been reached, this towel may be removed. When the distillation is completed the distillate is allowed to cool to room temperature and is then weighed in the flask. This weight minus that of the flask gives the weight of the total distillate.

The apparatus for freezing out and separating the paraffin scale is shown in Fig. 31. It consists of a bell jar about 16 centimeters high and 14 centimeters in diameter, surrounded by a felt or cotton cover, *b*. A copper jacket, *c*, 4½ centimeters in diameter at the top and 21 centimeters long, is held in the neck of the bell jar by means of a rubber stopper, *d*, and fits into the upper portion of the rubber stopper, *j*. A glass filter tube, *e*, fits inside the copper jacket, and to prevent circulation of air and condensation of water between it and the jacket a strip of heavy blotting paper, *f*, is wrapped around the top of the filter tube. Just below the constriction in the filter tube a wad of absorbent cotton, *g*, is placed, tightly compressed to a length of 2 centimeters by means of a glass rod. Above this is a wad of tightly packed asbestos wool, *h*, about 5 millimeters in length, upon which an asbestos filtering mat, *i*, is prepared. The filter tube passes through a rubber stopper, *j*, into a vacuum filtering flask, *k*, of 500 cubic centimeters capacity. The rubber stopper, *j*, is placed as tightly as possible against the neck of the bell jar, but to insure that there is no circulation of air a disk of blotting paper, *l*, is compressed between them. The thermometer, *m*, is capable of recording temperatures from  $-25^{\circ}\text{C.}$  to  $0^{\circ}\text{C.}$  and has the  $-20^{\circ}\text{C.}$  graduation at least 14 centimeters from the bulb. It is supported by means of



a guiding cork, *n*, and cork disk, *o*, which is held tightly against the top of the filter tube by the clamp, *p*.

The bell jar is filled with a freezing mixture of ice and salt in the proportion of three to one, which, as it melts, is drawn off through the bent glass tube, *g*, which is fitted with a rubber connection, *r*, and pinch cock, *s*, and collected in the 500 cubic centimeter filtering flask, *t*. The apparatus is supported on the ring, *u*, and condenser clamp, *v*, attached to the stand, *w*.

In separating the paraffin scale the following procedure is carried out. The filtering flask, *k*, is removed and a small cork stopper inserted in the lower end of the filter tube to assist in retaining the solution to be chilled in the upper part of the tube. Ten cubic centimeters of a mixture of equal parts Squibbs'

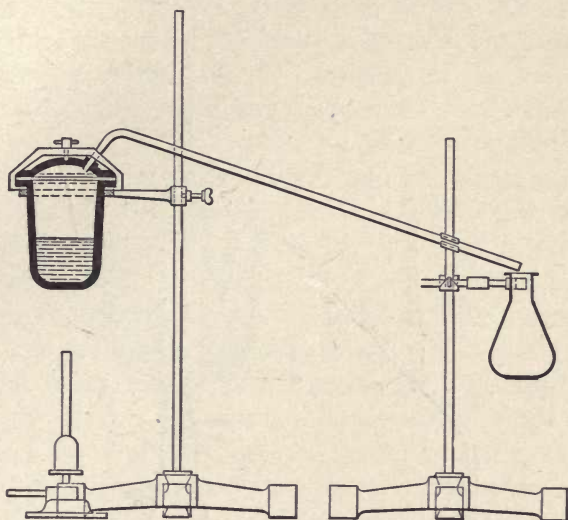


FIG. 30. Distillation Apparatus for Paraffin Scale Determination

ether and absolute alcohol is poured into the filter tube, the temperature of which has been reduced to  $-20^{\circ}\text{C}$ . From one to two grams of the well-mixed distillate obtained in the manner previously described is then accurately weighed in a 100 cubic centimeter Erlenmeyer flask, mixed with 10 cubic centimeters of Squibbs' ether, and poured into the filter tube. Ten cubic centimeters of absolute alcohol is next placed in the flask to wash out the ether solution, poured into the filter tube, and the cover carrying the thermometer placed on the tube. The mixture is maintained at a temperature  $-20^{\circ}\text{C}$ . for 15 minutes, then the cork stopper is removed from the outlet of the filter tube and the filtering flask is replaced. The corks supporting the thermometer are now loosened and a strong suction is applied to the filter flask until all of the solvent is drawn off. The contents of the filter tube are next washed with 10 cubic centimeters of a 1 to 1 mixture of Squibbs' ether and absolute alcohol, which is chilled to  $-20^{\circ}\text{C}$ . in the filter tube before suction is applied. When the washings have been removed the vacuum is turned off and the filter tube removed from the apparatus. The filter tube is then placed in a clean filter flask which also contains a 6-inch test tube in which the dissolved paraffin scale is later collected. About 10 cubic centimeters of warm petroleum ether are poured into the filter tube and allowed to remain until the paraffin scale has been dissolved. Vacuum is then applied and the dissolved

scale drawn into the test tube. This treatment is followed by two washings, one of 10 cubic centimeters and the other of 5 cubic centimeters of warm petroleum ether, which removes the last traces of paraffin scale. The entire contents of the test tube are then poured into a weighed platinum or glass crystallizing dish and the petroleum ether evaporated off over a steam bath. The dish is then placed in a drying oven maintained at 105° C. until the last

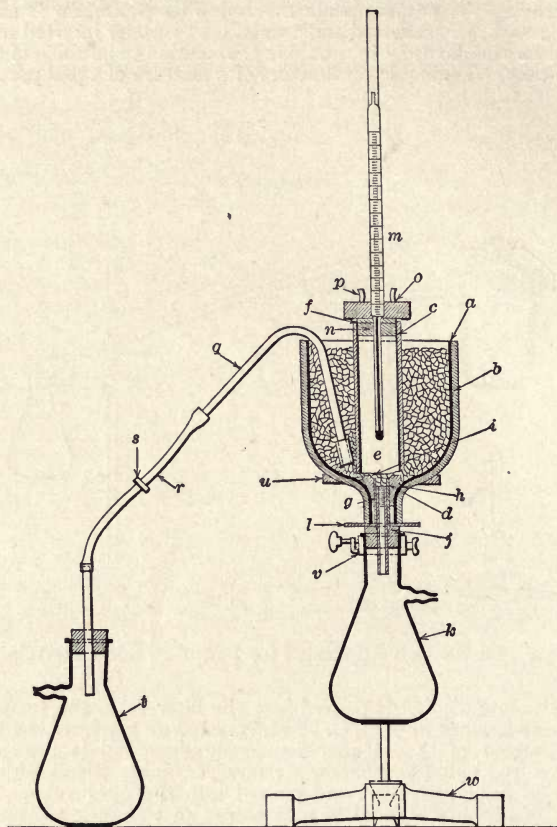


FIG. 31. Filtration Apparatus for Paraffin Scale Determination

traces of petroleum ether have been removed and the paraffin scale has attained a constant weight, after cooling in a desiccator.

The weight of the paraffin scale so obtained divided by the weight of the distillate taken and multiplied by the percentage of the total distillate obtained from the original sample equals the percentage of the paraffin scale. Thus, if 50 grams of the material produces 30 grams of distillate and this distillate yields 2.0 per cent paraffin scale, the percentage of paraffin scale upon the basis of the original sample would be as follows:

$$\text{Per Cent Paraffin Scale} = \frac{30}{50} \times 2\% = 1.2\%$$

*Value of Determination*

When made, this determination is confined to petroleum and asphalt products. It is not a thoroughly reliable quantitative determination, as unavoidable slight variations in the method and rate of distillation cause wide variations in results. Moreover, it appears likely that solid paraffins may be both destroyed and formed during the process of destructive distillation.

Owing to the fact that it is subject to wide variations it is of doubtful value except as a very roughly quantitative test for the purpose of identification. By some the presence of solid paraffins in a bituminous material for use in highway engineering is believed to be undesirable. While it is true that those petroleum products in which paraffin hydrocarbons greatly predominate are unsuited for use as cementing mediums, and while a high percentage of liquid paraffin hydrocarbons is often indicated by the presence of an appreciable amount of paraffin scale, it does not follow that the latter are themselves entirely undesirable constituents. In fact, if the material in which they occur possesses the requisite degree of cementitiousness, the presence of solid paraffins may make the material more chemically stable than it otherwise would be. There appears to be a general tendency to eliminate the paraffin scale clause in specifications for bituminous materials for the reason above stated. Most asphaltic petroleums and native asphalts contain only traces of paraffin scale, while the semi-asphaltic type shows comparatively small amounts.

## SPECIAL TESTS FOR EMULSIONS

The exact determination of the constituents of a bituminous emulsion is usually attended with considerable difficulty and no predetermined scheme can be made applicable to all materials of this character. The following methods, however, are used by the United States Office of Public Roads and Rural Engineering and have yielded satisfactory and fairly accurate results:



*Fatty and Resin Acids*

In order to break up the emulsion, a 20-gram sample is digested on a steam bath with 100 cubic centimeters of ( $\frac{N}{2}$  alcoholic potash). The digestion is carried out in a flask with a reflux condenser for about 45 minutes. The solution is filtered and the precipitate washed with 95 per cent alcohol. The filtrate is evaporated to dryness, after which the residue is taken up with hot water and any insoluble matter is filtered off. The aqueous solution, which contains the potassium soaps of the fatty acids, is acidified with dilute sulphuric acid and then shaken in a separatory funnel with petroleum ether. The aqueous portion is drawn off and the ethereal layer shaken up with cold water and washed twice, after which it is evaporated in a weighed platinum or porcelain dish to constant weight, first over a steam bath and then in a drying oven at 105° C. The residue consists of the fatty and resin acids present in the emulsion.

*Water*

The percentage of water in the emulsion is determined by distilling a 100-gram sample in the retort used for dehydration. The distillation is carried out in exactly the same manner as described under this test until the volume of water in the receiver shows no further increase. Any oils that come over are thoroughly mixed with the material remaining in the retort.

*Ammonia*

Many emulsions contain ammonia, and when this is present a second distillation of the material is necessary. This is carried out on a 100-gram sample in exactly the same manner as described for the determination of water, except for the fact that 40 cubic centimeters of a 10 per cent solution of caustic potash is added to the contents of the retort before beginning the distillation. The distillate is collected in a measured volume of  $\frac{N}{2}$  sulphuric acid. When the distillation is completed the excess of acid is titrated with  $\frac{N}{2}$  caustic potash, and the ammonia thus determined.

*Ash*

A one-gram sample of the dehydrated material is ignited in a weighed platinum or porcelain crucible. The ash will contain any inorganic matter from the bitumen as well as the fixed alkali present in the soap. The results are, of course, all calculated on the basis of the original material.

*Total Bitumen*

A two-gram sample of dehydrated material is extracted with carbon disulphide as described in the method for the determination of total bitumen, flask method, and in this manner the organic matter insoluble in carbon disulphide can be determined.

Having determined all constituents as above noted, it is assumed that the difference between their sum and 100 per cent is bitumen, which amount is reported accordingly.

## SOLUBILITY IN BENZOL OR CHLOROFORM

*Hot Extraction*

A hot extraction of a bituminous material may be made with any volatile solvent but, with the exception of creosoting oils and to some extent bituminous aggregates, cold extractions or those made at room temperature as de-

scribed under the determinations for total bitumen, asphaltenes and carbenes, are in more general use for materials directly used in highway engineering. While the total bitumen determination is for many reasons to be preferred as a single solvent test for creosoting oils, solvents other than carbon disulphide have been used for so long a time that most specifications for creosoting oils fail to include their solubility in carbon disulphide and require a test with benzol or chloroform, or both.

There are many types of hot extraction apparatus and a number of variations in methods of making the extraction. All of the hot extraction methods are, however, the same in general principle, and the following method substantially as recommended in 1915 by the Special Committee on Road Materials of The American Society of Civil Engineers may be considered as illustrative. Fig. 32 shows one type of extraction apparatus. In this apparatus the solvent is continuously boiled in the bottom of the glass receptacle and condensed at the top. The material to be extracted is held above the main mass of solvent in a filter suspended in a wire frame, but during the test remains in an atmosphere of the solvent vapor and is acted upon by the condensed portions of the solvent in finding their way back to the main mass. The solvent is thus used over and over while the soluble portion accumulates in the bottom of the receptacle.

From 5 to 10 grams of the water-free oil is weighed out into a weighed 100-c.c. beaker. Fifty c.c. of the solvent are added, and the solution is passed through a weighted 9-cm. C. S. & S. No. 575 filter paper in a short-stemmed funnel or an alundum crucible, the filtrate being passed into the flask to be subsequently used for the hot extraction. The beaker is washed clean from all soluble matter, dried, and weighed. The filter paper or crucible and contents are then placed in the extraction apparatus, and heat is applied from a water bath or hot plate until the extraction is complete and the filtrate runs through colorless. The filter and contents are then dried in a hot-air oven at about 90°C., cooled in a desiccator, and weighed. The increase in weight is added to the increase in weight of the beaker, if any, the result being the weight of the insoluble matter. The weight of the insoluble matter thus found is subtracted from the weight of the material taken for analysis. The difference in weight is the weight of the soluble matter, from which the percentage is calculated. If a filter paper is used in this test it is advisable to first soak it in the solvent to remove any grease which it may hold and then dry, cool and weigh it in exactly the same manner as will be used after the extraction is complete.

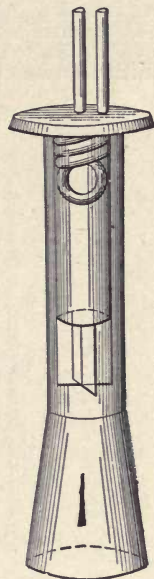


FIG. 32. Hot Extractor

#### SPECIAL TESTS FOR CREOSOTING OILS

The following tests are slightly modified from descriptions by Church.\* They are roughly quantitative and are not susceptible to a great degree of refinement.

\* Methods for Testing Coal Tar and Refined Tars, Oils and Pitches Derived Therefrom. *Jour. Ind. and Eng. Chem.* Vol. 3, p. 227.



*Tar Acids*

One hundred cubic centimeters of the material are weighed into an Engler distilling flask, which is then set up as shown in Fig. 33. Heat is applied so that the distillate comes over at the rate of about 5 cubic centimeters per minute. Distillation may be stopped when the thermometer reaches  $355^{\circ}\text{C}$ . When distilling distillate oils, Church carries the distillation to a point where at least 95 per cent has distilled off. The condenser tube is kept sufficiently warm, by means of a burner if necessary, to prevent solidification of the distillate. The total distillate is collected in a special graduated form of separatory funnel.

After distillation the separatory funnel and contents are warmed in water to  $60^{\circ}\text{C}$ . and the volume noted. Fifty cubic centimeters of a ten per cent caustic soda solution are then added and the whole well shaken. The mixture

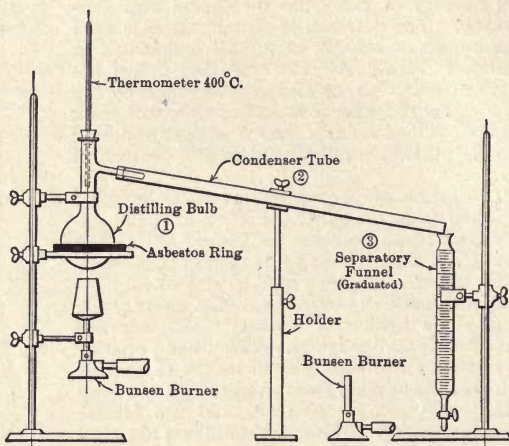


FIG. 33. Distillation Apparatus for Determination of Tar Acids

is then allowed to settle, the clear soda solution drawn off and the contents of the funnel again warmed to  $60^{\circ}\text{C}$ . and the volume is read. If a shrinkage from the first reading is noted, 30 cubic centimeters of the soda solution are added and the operation repeated until no further shrinkage in volume is noted between the last two readings. The total shrinkage in cubic centimeters divided by the original volume of distillate is taken as the percentage of tar acids in the distillate. This percentage may then be figured upon the basis of the original material by correcting the original recorded volume of the distillate to normal temperature. For this purpose the original volume of distillate may be divided by 1.028.

*Dry Naphthalene*

The extracted distillate from the determination of tar acids is placed in a copper beaker and maintained at  $15.5^{\circ}\text{C}$ . for 15 minutes. The material is then filtered in a funnel fitted with a filter cone and vacuum applied until no more oil is removed. Any naphthalene in the filter is then pressed between paper in a letter press to remove as much of the absorbed oil as possible, after which it is weighed and its percentage figured on the weight of original material which was distilled in the determination of tar acids.



*Sulphonation Test*

Two hundred cubic centimeters of the material is weighed into a 500-cc. Jena flask, which is then set up as shown in Fig. 34. Distillation is carried to 320° C. and the distillate from 305° to 320° C. is caught in a tared receiver and its weight ascertained. The weighed fraction is then warmed to 60° C. with from four to five times its volume of concentrated sulphuric acid, and the whole transferred to a special graduated form of separatory funnel. (See Fig. 22.) The receiver is rinsed three times with concentrated sulphuric acid and the rinsings added to the contents of the funnel.

The funnel is then stoppered and shaken, first cautiously, then vigorously, for about 15 minutes and the contents allowed to settle overnight. The acid is then carefully drawn down into the graduated portion of the funnel to within

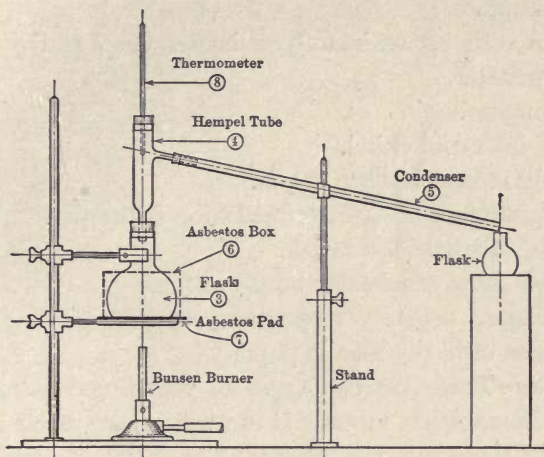


FIG. 34. Distillation Apparatus for Sulphonation Test

2 cubic centimeters of any unsulphonated residue. Whether or not any unsulphonated residue is visible, the test is carried further by adding about 20 cubic centimeters of water and allowing it to settle for one half-hour. The water is then drawn down as close as possible without removing any supernatant oil or emulsion. Ten cubic centimeters of strong sulphuric acid are next added and allowed to settle for from 15 to 20 minutes. Any unsulphonated residue should now settle out clear and give a distinct reading. If it amounts to less than 0.2 cubic centimeter it should be drawn down into the narrow part of the funnel just above the stopcock, where it may be estimated to 0.01 cubic centimeter. The number of cubic centimeters is figured as percentages on the weight of the fraction taken. Thus if the original fraction weighs 10 grams and 0.5 cubic centimeter of unsulphonated residue remains, this residue is reported as 5 per cent of the fraction.

If the unsulphonated oil is dark in color it should be treated with an excess of 10 per cent sodium hydroxide solution. If the oil is soluble in this reagent the test is regarded as negligible.

## EXTRACTION OF BITUMINOUS AGGREGATES AND RECOVERY OF BITUMEN AND AGGREGATE

### CENTRIFUGAL EXTRACTION

#### *Equipment:*

- 1 centrifuge extractor, complete with motor, speed regulator, and electrical connections.
- 1 hot plate.
- 1 tin dish approximately 2 inches deep and 9 inches in diameter.
- 1 hammer.
- 1  $\frac{3}{4}$ -inch cold chisel.
- 1 large metal kitchen spoon.
- 1 square foot of  $\frac{1}{16}$ -inch deadening felt paper.
- 1  $1\frac{1}{2}$ -inch stiff flat brush.
- 1 500 cubic centimeter bottle or flask.
- 1 balance, capacity 1 kilogram, sensitive to 0.1 gram.
- 1 sheet of heavy manila paper.

*Method.*—There are two types of centrifugal extractors for bituminous materials in general use which are similar in design, the Reeve type and the Dulin. The latter is made in two sizes, one primarily for coarse aggregates and the other for the extraction of fine aggregates such as sheet asphalt topping when it is desired to recover the mineral matter for further test. The former, shown in Fig. 35, was designed upon lines suggested by an examination of machines in use by A. E. Schutte and C. N. Forrest. It consists of a  $\frac{1}{5}$ -horsepower, 1100 revolutions per minute, vertical-shaft, electric motor, *a*, with the shaft projecting into the cylindrical copper box, *b*, the bottom of which is so inclined as to drain to the spout, *c*. A  $\frac{3}{16}$ -inch circular brass plate  $9\frac{1}{2}$  inches in diameter is shown in *d*, and upon this rests the sheet-iron bowl, *e*, which is  $8\frac{1}{2}$  inches in diameter by  $2\frac{5}{16}$  inches high, and has a 2-inch circular hole in the top. Fastened to the inner side of the bowl is the brass cup, *f*, having a circle of  $\frac{1}{8}$ -inch holes.

for the admission of the solvent, and terminating in the hollow axle, which fits snugly through a hole at the center of the brass plate. The bowl may be drawn firmly against a felt-paper ring, *g*,  $\frac{3}{4}$  inch wide, by means of the  $2\frac{1}{2}$ -inch milled nut, *h*, for which the hollow axle is threaded for a distance of  $\frac{3}{4}$  inch directly below the upper surface of the plate. The axle fits snugly over the shaft of the motor, to which it is locked by a slot and cross pin *i*.

The aggregate is prepared for analysis by heating it in a tin dish on the hot plate until it is sufficiently soft to be thoroughly

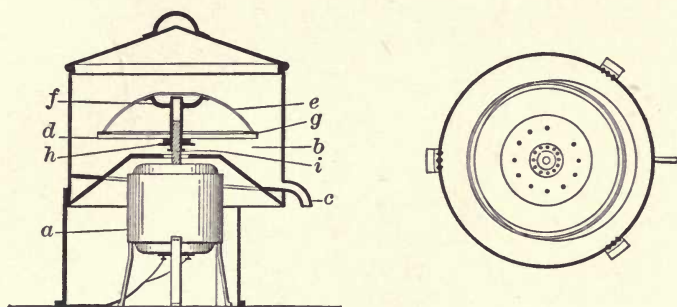


FIG. 35. Centrifugal Extractor  
(Reeve Type)

disintegrated by means of a large spoon. Care must be taken, however, that the individual particles are not crushed. If a section of pavement is under examination, a piece weighing somewhat over 1 kilogram may be cut off with hammer and chisel. The disintegrated aggregate is then allowed to cool, after which a sufficient amount is taken to yield on extraction from 50 to 60 grams of bitumen. It is placed in the iron bowl and a ring  $\frac{3}{4}$  inch wide, cut from the felt paper, is fitted on the rim, after which the brass plate is placed in position and drawn down tightly by means of the milled nut. If the bitumen is to be recovered and examined, the felt ring should be previously treated in the empty extractor with a couple of charges of carbon disulphide in order to remove any small amount of grease or resin that may be present, although a



proper grade of felt should be practically free from such products. The bowl is now placed on the motor shaft and the slot and pin are carefully locked. An empty bottle is placed under the spout and 150 cubic centimeters of carbon disulphide are poured into the bowl through the small holes. The cover is put on the copper box and, after allowing the material to digest for a few minutes, the motor is started, slowly at first in order to permit the aggregate to distribute uniformly. The speed should then be increased sufficiently by means of the regulator

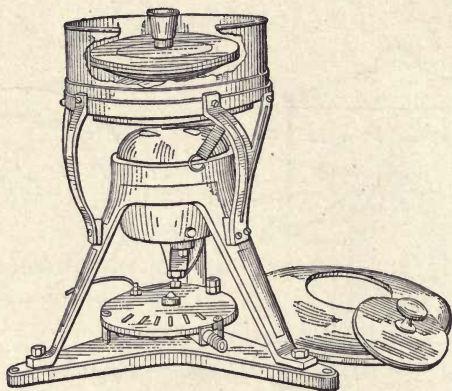


FIG. 36. Dulin Rotarex  
(Small Size)

to cause the dissolved bitumen to flow from the spout in a thin stream. When the first charge has drained, the motor is stopped and a fresh portion of disulphide is added. This operation is repeated from four to six times with 150 cubic centimeters of disulphide.

With a little experience the operator can soon gauge exactly what treatment is necessary for any given material. When the last addition of solvent has drained off, the bowl is removed and placed with the brass plate uppermost on a sheet of manila paper. The brass plate and felt ring are carefully laid aside on the paper and, when the aggregate is thoroughly dry, it can be brushed on a pan of the rough balance and weighed. The difference between this weight and the original weight taken

shows the amount of bitumen extracted. The aggregate may then be tested as occasion requires.

The operation of the Dulin Rotarex shown in Fig. 36 is practically identical with that above described.

## HOT EXTRACTION

*Equipment.*—Same as for centrifugal extraction except that a New York Testing Laboratory hot extractor, complete with electric-light bulb, electrical and cold-water connections, replaces the centrifugal extractor.

*Method.*—The New York Testing Laboratory extractor shown in Fig. 37 consists of a large brass cylinder, through the bottom of which projects a 16-candle-power incandescent carbon-filament

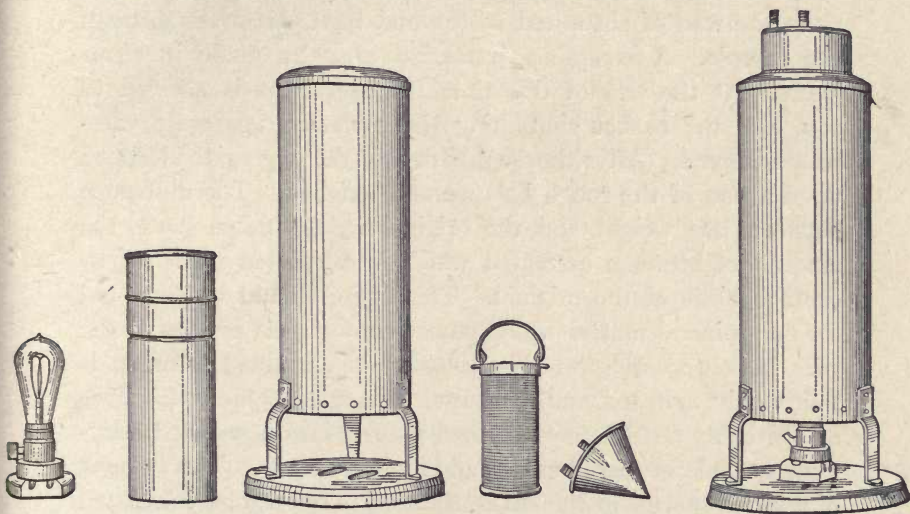


FIG. 37. New York Testing Laboratory Hot Extractor

bulb to supply heat to the extraction apparatus proper, which is held in the upper portion of the cylinder. This apparatus is composed of a cylindrical brass vessel for holding the solvent, a cylindrical wire basket made of 80-mesh wire cloth, suspended in the cylinder, and an inverted conical condenser which serves as a top.



The aggregate is prepared for analysis by heating it in a tin dish on the hot plate until it is sufficiently soft to be disintegrated by means of a large spoon. The disintegrated aggregate is then allowed to cool. Five hundred grams of aggregates containing particles larger than  $\frac{1}{2}$  inch in diameter and three hundred grams of aggregates with all particles smaller than  $\frac{1}{2}$  inch are then closely packed in the wire basket and covered with a disk or wad of absorbent cotton or felt. From 175 to 200 cubic centimeters of carbon disulphide are next placed in the inside vessel, in which the wire basket should be suspended. The top is then placed in position and cooling water circulated through it. Heat is applied by means of the electric-light bulb. The solvent is boiled in the lower part of the extractor, and condenses on the under surface of the top, from which it drips upon the wad of absorbent cotton and then percolates through the sample. A complete extraction may be made in three hours. At the end of this time the apparatus is allowed to cool and the basket containing the extracted aggregate carefully removed. After thoroughly drying the aggregate is placed upon a pan of the rough balance and weighed. The difference between this weight and the original weight taken shows the amount of bitumen extracted which is calculated upon a percentage basis of the original. This figure should be corrected for fine mineral matter which passes through the meshes of the wire basket as follows: The solution of extracted bitumen is thoroughly agitated and measured in a glass graduate. Five or ten cubic centimeters are then poured into a weighed platinum crucible or dish, burned, and ignited to ash. The amount of mineral matter in the entire solution may then be calculated from the amount of ash produced from that portion ignited. The total percentage of such ash is then deducted from the percentage of bitumen already calculated in order to obtain the true percentage of bitumen. The amount of this correction will ordinarily vary from 0.1 per cent in uniformly coarse aggregates to 1.0 or 2.0 per cent in the analysis of aggregates containing a considerable amount of very fine mineral matter.



## RECOVERY OF BITUMEN

If an extraction has been made upon a sufficiently large sample of bituminous aggregate to yield from 30 to 50 grams of pure bitumen, it may be desirable to recover and examine the bitumen. When this is so the apparatus shown in Fig. 38 will be found convenient and fairly safe for the distillation and recovery of such inflammable solvents as carbon disulphide. In the laboratory of the Office of Public Roads and Rural Engineering this apparatus is arranged so that the glass tubing passes through a stone partition between two sections of a small

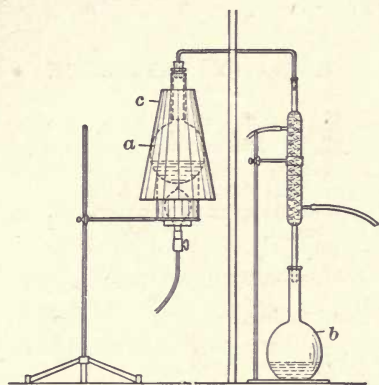


FIG. 38. Distillation Apparatus for Recovering Bitumen

hood, thus keeping the distilling and receiving apparatus entirely separated.

The solution of bitumen should be allowed to stand overnight in order to permit the settling of any fine mineral matter that is sometimes carried through the extractor. The solution is then decanted into the flask *a*, and the solvent is driven off by means of heat from an incandescent lamp until the residue is of a thick, sirupy consistency. Meanwhile the solvent is condensed and recovered in the flask *b*. The residue is poured into an 11-centimeter porcelain evaporating dish and evaporated on a steam bath. The most scrupulous care must be taken

at all times that no flames are in its immediate vicinity. Evaporation is carried on at a gentle heat, with continual stirring, until foaming practically ceases. It is advisable to have a large watch glass at hand to smother the flames quickly should the material ignite. As the foaming subsides, the heat of the steam bath may be gradually raised, and evaporation is continued until the bubbles beaten or stirred to the surface of the bitumen fail to give a blue flame or odor of sulphur dioxide when ignited by a small gas jet. The dish of bitumen should then be set in a hot-air oven maintained at  $105^{\circ}$  C. for about an hour, after which it is allowed to cool. Its general character is noted and any tests for bitumens that are necessary are then made upon it.

#### FILTRATION EXTRACTION

##### *Equipment:*

- I short stem funnel,  $2\frac{1}{2}$  inches in diameter.
- I 9-centimeter Schleicher & Schull 597 filter paper.
- I  $2\frac{1}{2}$ -inch watch glass.
- 3 250 cubic centimeter Erlenmeyer flasks.
- I piece glazed paper about 12 inches square.
- I small camel's-hair brush.
- I small platinum dish.
- I wash bottle for solvent.
- I hot-air oven.
- I Bunsen burner and tubing.
- I spatula.
- I nichrome triangle.
- I small metal pan.
- I analytical balance, capacity 100 grams, sensitive to 0.1 milligram.

*Method.*—When it is not desired to recover the bitumen in a bituminous aggregate containing no mineral particles coarser than sand, and a small centrifugal extractor is not at hand, the following method, substantially as described by Richardson,\* will be found useful.

The bituminous aggregate is first disintegrated by warming it in a small metal pan and then allowed to cool. A ten-gram sample is then weighed out upon a watch glass and carefully transferred to a Schleicher & Schull 597 filter paper folded in the funnel. The funnel is then placed in the mouth of a 250-cc. Erlenmeyer flask. A fine stream of carbon disulphide is directed on the surface of the aggregate until it is thoroughly saturated, but no excess of solvent is used. The moistened aggregate is then allowed to stand until it has softened and settled upon the filter. The filter is then filled with carbon disulphide to within an eighth of an inch of the top and the funnel covered with a  $2\frac{1}{2}$ -inch watch glass. As percolation proceeds, additional solvent is added, and filtration may be hastened by occasionally washing between the paper and funnel with the solvent. During the day the aggregate is washed as clean as possible, and both it and the filtrate are allowed to stand overnight.

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\*"The Modern Asphalt Pavement." John Wiley & Sons, p. 571.

The next morning the funnel is placed in a clean, dry flask, and the filtrate is carefully decanted into another flask without disturbing the sediment. A small portion of carbon disulphide is then poured into the first flask, shaken up, and poured upon the filter. Any remaining sediment is then washed from the flask upon the filter, a feather being used if necessary to thoroughly clean it. The mineral aggregate upon the filter is then washed until the filtrate runs colorless.

After thoroughly drying in air, the funnel and contents are placed in an oven at 105° C. for 30 minutes, after which it is separated from the filter over a piece of glazed paper by scraping with a blunt spatula and rubbing between the fingers, care being taken not to detach any fibers of the paper. It is then dusted into a tared watch glass and weighed.

Meanwhile the filtrate is poured into a weighed platinum dish and burned under a well-ventilated hood. If the dish is not sufficiently large to hold all at one time, it is burned in portions, but care must be taken that the dish and contents are thoroughly cool before adding a fresh portion. The dish is finally ignited and the filter paper placed in it and also ignited. It is then cooled and weighed. The combined weight of the contents of the watch glass and the platinum dish is then taken as the weight of mineral matter and the weight of bitumen ascertained by subtracting the combined weight from that of the original sample. The percentages of aggregate and bitumen are calculated upon a percentage basis of the whole. The recovered aggregate may then be subjected to a sieve analysis.

#### GRADING OF MINERAL AGGREGATES

##### *Equipment:*

- 1 set of 8-inch stone sieves with circular openings of  $1\frac{1}{2}$ ,  $1\frac{1}{4}$ , 1,  $\frac{3}{4}$ ,  $\frac{1}{2}$ , and  $\frac{1}{4}$  inches, respectively.
- 1 set of 8-inch brass sand sieves of 10, 20, 30, 40, 50, 80, 100, and 200 mesh, respectively, with pan and cover.
- 1 rough balance, capacity 1 kilogram, sensitive to 0.1 gram.
- 1  $1\frac{1}{2}$ -inch stiff, flat brush.

Several sheets of manila paper.

*Method.*—It is often desirable to make a mechanical analysis not only of an extracted aggregate but also of a mineral aggregate which is to be used in a bituminous mix. In the latter case almost any reasonable-size sample may be secured, but with extracted aggregates the size of the sample is limited by the amount of bituminous aggregate which it is practicable to obtain in a single extraction. In such cases it is customary to work with the entire mass of extracted aggregate and to consider all mineral matter obtained as a correction, by burning and igniting the filtrate, as material passing a 200-mesh sieve. Results may then be expressed on a percentage basis of the



original bituminous aggregate or upon the extracted aggregate as occasion may require. In reporting results the percentage of fine material is sometimes given first, each larger size following in order, while in other cases the opposite procedure is followed and the coarsest size reported first.

For aggregates containing particles too large to pass a 10-mesh screen, the stone sieves are used, and are stacked in their regular order over a sheet of heavy paper, with the largest size required on top. The weighed amount of stone is placed on the largest sieve and is carefully protected from drafts which might carry away any of the fine material. The upper sieve is then removed from the stack and shaken over a large sheet of paper until no more particles come through. The material thus retained, including any fragments caught in the meshes of the sieve, is weighed and that which passes is added to the contents of the succeeding sieve. This operation is repeated with each succeeding sieve.

When grading sands or fine aggregates, it is customary to take a 100-gram sample in order that the weights may give direct percentages to tenths of one per cent. The sieves are stacked in regular order, with the 200-mesh sieve resting on the pan. The sample is brushed on the top sieve, after which the cover is put on and the stack agitated for about five minutes with both rocking and circular shaking. Each sieve is removed in order, and shaken and tapped on a clean piece of paper until no appreciable amount of material comes through. All lumps are broken up by crushing them against the side of the sieve with the finger or a small spatula. The contents of the sieve are emptied into the pan of the balance. All particles caught in the mesh are removed by brushing across the under side of the sieve and are added to the contents of the pan. As great opportunity exists for wide variations in the results of sand gradings made by different persons, owing to the possibility of always getting a little more material to pass by continued shaking, it is well for the novice to repeat his sifting on any given mesh, after having weighed it, in order to see what further loss he can produce. If his judgment has

not erred, several minutes' further sifting should not produce a loss of over 0.5 gram.

Where coarse aggregates have considerable material passing a 10-mesh sieve and it is desired to grade this material further it should be weighed and well mixed, quartered, if necessary, and a 100-gram sample should be passed through the sand sieves. From the percentages so obtained and the weight of material passing the 10-mesh sieve, the percentages of the total aggregate which these finer materials represent may be calculated.

In the above operations, screens and sieves recommended in 1916 by Committee D-4 of The American Society for Testing Materials should be used. Following are given methods for making a mechanical analysis of three types of mineral aggregate as recommended in 1916 by the Committee:

*Proposed Revised Standard Method  
for*

*Making a Mechanical Analysis of Sand or Other Fine Highway  
Material, Except for Fine Aggregate Used in Cement Concrete*

The method shall consist of (1) drying at not over 110° C. (230° F.) to a constant weight a sample weighing 50 g.; (2) passing the sample through each of the following mesh sieves (American Society for Testing Materials standard sieves):\*

Meshes per Linear Inch (= 2.54 cm.)	DIAMETER OF WIRE	
	In.	Mm.
10.....	0.027	0.6858
20.....	0.0165	0.4191
30.....	0.01375	0.34925
40.....	0.01025	0.26035
50.....	0.009	0.22865
80.....	0.00575	0.1460
100.....	0.0045	0.1143
200.....	0.00235	0.05969

\* The order in which the sieves are to be used in the process of sifting is immaterial and may be left optional; but in reporting results the order in which the sieves have been used shall be stated.

(3) determining the percentage by weight retained on each sieve, the sifting being continued on each sieve until less than 1 per cent of the weight retained on each sieve shall pass through the sieve during the last minute of sifting, and  
 (4) recording the mechanical analysis in the following manner:

Passing 200-mesh sieve.....	=	.....	per cent
Passing 100-mesh sieve and retained on a 200-mesh sieve =	.....	"	
Passing 80-mesh sieve and retained on a 100-mesh sieve =	.....	"	
Passing 50-mesh sieve and retained on an 80-mesh sieve =	.....	"	
.....	=	.....	"
		100.00	"

### *Proposed Standard Method*

*for*

#### *Making a Mechanical Analysis of Broken Stone or Broken Slag, Except for Aggregates Used in Cement Concrete*

The method shall consist of (1) drying at not over 110° C. (230° F.) to a constant weight a sample weighing in pounds six times the diameter in inches of the largest holes required; (2) passing the sample through such of the following size screens having circular openings as are required or called for by the specifications, screens to be used in the order named: 8.89 cm. (3½ in.), 7.62 cm. (3 in.), 6.35 cm. (2½ in.), 5.08 cm. (2 in.), 3.81 cm. (1½ in.), 3.18 cm. (1¼ in.), 2.54 cm. (1 in.), 1.90 cm. (¾ in.), 1.27 cm. (½ in.), and 0.64 cm. (¼ in.); (3) determining the percentage by weight retained on each screen, and  
 (4) recording the mechanical analysis in the following manner:

Passing 0.64-cm. (¼-in.) screen.....	=	.....	per cent
Passing 1.27-cm. (½-in.) screen and retained on a 0.64-cm. (¼-inch) screen.....	=	.....	"
Passing 1.90-cm. (¾-in.) screen and retained on a 1.27-cm. (½-in.) screen.....	=	.....	"
Passing 2.54-cm. (1-in.) screen and retained on a 1.90-cm. (¾-in.) screen.....	=	.....	"
		100.00	"

### *Proposed Standard Method*

*for*

#### *Making a Mechanical Analysis of Mixtures of Sand or Other Fine Material with Broken Stone or Broken Slag, Except for Aggregates Used in Cement Concrete*

The method shall consist of (1) drying at not over 110° C. (230° F.) to a constant weight a sample weighing in pounds six times the diameter in inches of the largest holes required; (2) separating the sample by the use of a screen having circular openings 0.64 cm. (¼ in.) in diameter; (3) examining the portion retained on the screen in accordance with the proposed Standard Method for Making a Mechanical Analysis of Broken Stone or Broken Slag, Except for Aggregates Used in Cement Concrete; (4) examining the portion passing this screen in accordance with the proposed Standard Method for Making a Mechanical Analysis of Sand or Other Fine Highway Material, Except for Fine Aggregate Used in Cement Concrete, and (5) recording the mechanical analysis in the following manner:



Passing 200-mesh sieve.....	=	.....	per cent
Passing 100-mesh sieve and retained on a 200-mesh sieve	=	.....	"
Passing 80-mesh sieve and retained on a 100-mesh sieve	=	.....	"
.....	=	.....	"
Passing 10-mesh sieve and retained on a 20-mesh sieve...	=	.....	"
Passing 0.64-cm. ( $\frac{1}{4}$ -in.) screen and retained on a 10-mesh sieve.....	=	.....	"
Passing 1.27-cm. ( $\frac{1}{2}$ -in.) screen and retained on a 0.64-cm. ( $\frac{1}{4}$ -in.) screen.....	=	.....	"
Passing 1.90-cm. ( $\frac{3}{4}$ -in.) screen and retained on a 1.27-cm. ( $\frac{1}{2}$ -in.) screen.....	=	.....	"
.....	=	.....	"
			<hr/>
			100.00
			"

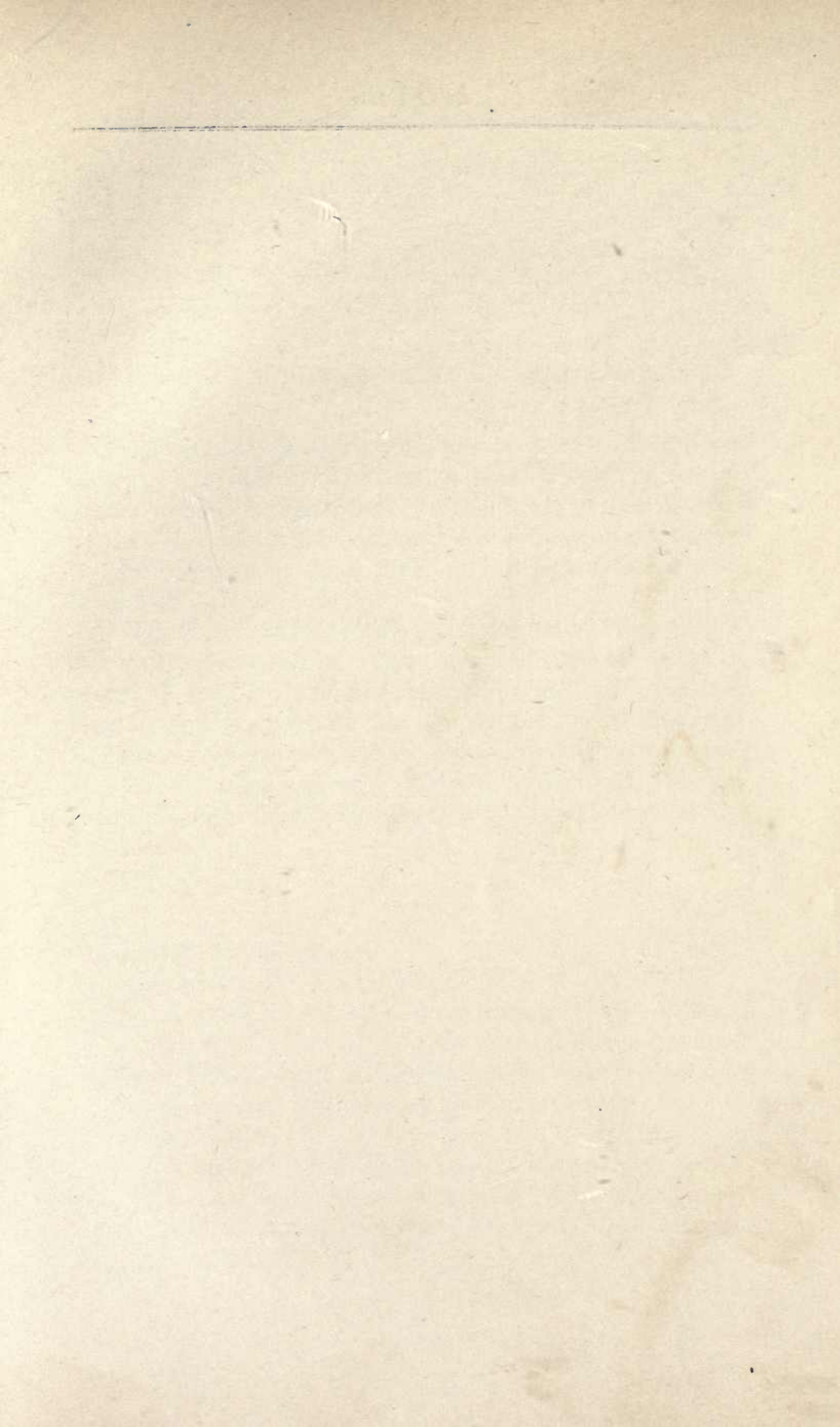
# VALUE OF TESTS FOR BITUMINOUS AGGREGATES

The determination of bitumen in bituminous aggregate is particularly valuable as a laboratory control test for the plant or factory manufacture of such aggregates. The serviceability of a bituminous aggregate is not only dependent upon the physical and chemical properties of the bituminous cement, but also upon the percentage of such cement which is present. Too much may be almost as bad as too little, and the exact percentage should therefore be adjusted to meet the character and grading of the mineral aggregate which is used. When once the proper combination of bituminous cement and a given aggregate has been experimentally ascertained, the per cent of bitumen as determined by test should, for different lots of the same bituminous aggregate, fall within comparatively narrow limits.

While in the determination of total bitumen or those materials consisting mainly of bitumen it is highly desirable that for the sake of uniformity all determinations be made with the cold solvent, the same is not so important in the case of bituminous aggregates. Hot and cold methods of extraction are, therefore, equally serviceable for the latter, and the results obtained by both methods are ordinarily quite comparable and to all extents and purposes equivalent. It is well known that many bituminous materials are more soluble in a hot solvent than in the same solvent cold. The actual difference is necessarily greater in materials rich in bitumen than for those comparatively poor in the same bitumen. Thus a given type of

relatively pure bitumen may, for the sake of illustration, be considered as being as much as 2 per cent more soluble in a hot solvent than in the same solvent cold. If this same bitumen were, however, mixed with nine times its weight of mineral matter, it will be seen that the difference in the actual percentage of bitumen determined by hot and cold extractions will amount to not over 0.2 per cent.

In the extraction of bituminous aggregates it is frequently desirable to recover and examine both the extracted bitumen and the mineral aggregate. The bitumen may thus often be identified and its suitability as a cementing medium ascertained. The mechanical grading of the aggregate will also indicate the suitability of the mixture for a specific purpose, provided the characteristics of the bituminous cement are known. If the specific gravity of both bitumen and mineral matter are determined as well as the relative proportions in which each occur, it will be possible to ascertain the rational per cent of bitumen in the mixture and also its theoretical maximum density as described under "Bituminous Aggregates," in Part III.





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### PART III. CHARACTERISTICS OF THE MORE IMPORTANT BITUMINOUS MATERIALS

#### FLUID PETROLEUM PRODUCTS AND EMULSIONS

Fluid petroleum products are used as dust preventives, fluxes, carpeting mediums, and occasionally, when very viscous, as bituminous cements for certain types of mineral aggregates. Emulsifying oils and petroleum emulsions are used as dust preventives or light carpeting mediums, while asphalt emulsions are sometimes used as bituminous cements.

Almost any sufficiently fluid petroleum product may be used as a dust preventive and, unless successive applications are expected to build up a bituminous carpet, no tests other than viscosity and relatively low loss by volatilization are important. If, however, the material is to serve as a carpeting medium, other tests are of value, and as a rule all of the fluid petroleum products may ordinarily be subjected to the following tests to advantage:

#### ORDINARY TESTS

1. Specific Gravity  $25^{\circ}/25^{\circ}$  C.
2. Specific Viscosity, Engler, 50 c.c.
  - a. Preferably at  $25^{\circ}$  C. if the material is to be used cold.
  - b. At  $50^{\circ}$  C. or  $100^{\circ}$  C. if the material is to be used hot.
3. Float Test at  $50^{\circ}$  C. if the material is to be used primarily as a carpeting medium or a bituminous cement.
4. Flash Point  $^{\circ}$  C.
  - a. Open-cup method for non-volatile products.
  - b. Closed-cup method for volatile, crude, topped or cut-back products, particularly if they are to be applied hot.



5. Volatilization Test, 50 grams, 5 hours.
  - a. At  $100^{\circ}$  C. for cut-back products which are required to harden rapidly after use.
  - b. At  $163^{\circ}$  C. for practically all products.
6. Consistency of Residue from Volatilization Test for carpeting mediums and bituminous cements.
  - a. Float Test at  $50^{\circ}$  C. if too soft for penetration test.
  - b. Penetration Test  $25^{\circ}$  C., 100 grams, 5 seconds, when possible.
7. Total Bitumen.
  - Organic Matter Insoluble in Carbon Disulphide.
  - Inorganic Matter Insoluble in Carbon Disulphide.
8. Asphaltenes.
9. Carbenes on cut-back products.
10. Fixed carbon when possible.

## INTERPRETATION OF RESULTS IN TABLE I

Table I, on page 123, shows the results of the above tests upon typical fluid petroleum products of interest in highway engineering.

The heavy distillate indicated by its fairly high specific gravity should be considered merely as a dust preventive. This product is very fluid, as shown by its low specific viscosity at  $25^{\circ}$  C., and is therefore susceptible to application by means of an ordinary gravity water sprinkler. Its flash point is relatively high for a distillate, and this, together with a loss at  $163^{\circ}$  C., of only 12.6 per cent, indicates that it would not volatilize too rapidly after application. The fact that the residue from the volatilization test is fluid shows that it will develop no appreciable binding value after application, and this, together with a total absence of asphaltenes and practically no fixed carbon, shows it to be a permanent lubricant which should be used sparingly so as not to disintegrate the road surface. Being a bituminous distillate, it is naturally entirely soluble in carbon disulphide.

The medium crude petroleum is of Mexican origin, its gravity being considerably lower than the maltha. Its viscosity at



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TABLE I  
ANALYSES OF FLUID PETROLEUM PRODUCTS

Test	Heavy Distillate	Medium Crude Asphaltic	RESIDUALS			Maltha	Cut-Back Asphalt
			Paraffin	Semi-Asph.	Asphaltic		
Sp. Gr. 25°/25° C.....	0.892	0.949	0.936	0.971	0.990	0.982	0.999
Sp. Vis., Engler, 50 c.c., 25° C.....	3.4	94.8	.....	.....	.....	.....	.....
Sp. Vis., Engler, 50 c.c., 50° C.....	.....	18.2	42.3	.....	.....	.....	.....
Sp. Vis., Engler, 50 c.c., 100° C.....	.....	.....	.....	15.0	22.7	13.5	.....
Float Test, 50° C.....	.....	.....	.....	42"	1' 12"	30"	3' 49"
Flash Point, closed cup.....	105° C.	30° C.	.....	.....	.....	35° C.	.....
Flash Point, open cup.....	.....	.....	205° C.	245° C.	167° C.	.....	200° C.
Loss, 163° C., 5 hrs., 50 grams.....	12.6 %	30.5 %	1.0 %	0.1 %	1.3 %	17.7 %	5.7 %
Float Test, Residue, 50° C.....	Fluid	1' 23"	Fluid	47"	1' 37"	8' 28"	.....
Penetra. Res., 25° C., 100 g., 5 sec.....	.....	.....	.....	.....	.....	139	98
Total Bitumen (Sol. in CS <sub>2</sub> ).....	100.00 %	99.90 %	99.92 %	99.91 %	99.92 %	99.96 %	99.83 %
Organic matter insoluble.....	0.00	0.05	0.05	0.05	0.06	0.04	0.13
Inorganic matter insoluble.....	0.00	0.05	0.03	0.04	0.02	0.00	0.04
Bit. insol. 86° B. naph. (Asphaltenes).....	100.00 %	100.00 %	100.00 %	100.00 %	100.00 %	100.00 %	100.00 %
Carbenes.....	0.0 %	14.1 %	2.0 %	7.7 %	6.2 %	20.2 %	25.3 %
Fixed Carbon.....	0.4 %	7.7 %	0.0 %	0.0 %	0.0 %	0.0 %	0.0 %
			3.2 %	5.6 %	6.4 %	7.8 %	12.2 %



25° C. shows it to be fairly fluid and susceptible to cold application, although at as high as 50° C. it is more viscous than the distillate. Its very low flash point and high loss at 163° C. shows it to contain a large amount of volatile products. The fact that a float test of 1' 23" is obtained at 50° C. upon the residue, indicates a tendency to harden to a point at which it may serve as a weak binding medium. Its asphaltic nature is indicated by its relatively high percentages of asphaltenes and fixed carbon. Like the other products it is practically pure bitumen, being almost completely soluble in carbon disulphide. Such a material may be considered as other than a mere dust preventive, as it may be used to build up a very thin bituminous carpet. Unless used sparingly, however, because of the fact that it hardens very slowly, it will tend to emulsify on the road surface in wet weather and produce a disagreeable mud.

The three residual petroleum products may be considered together. It will be noted that the viscosity of all are too high to permit of their application to a road surface without first heating them to a state of considerably greater fluidity. Their relatively high flash point and low loss by volatilization at 163° C. show them to be residuals and not crude products. They are all practically completely and equally soluble in carbon disulphide and carbon tetrachloride, which shows that they have not been injuriously cracked in their manufacture. The residue from the volatilization test of the paraffin residuum is too fluid for a float test at 50° C., which, together with the very low percentage of asphaltenes and fixed carbon, shows the material to be absolutely unsuited for use as a carpeting medium. As it is too viscous to apply cold it would also be an unsuitable dust preventive. The semi-asphaltic residuum which is a Texas product is sufficiently viscous to show an appreciable float test at 50° C., but is incapable of hardening materially as shown by its almost negligible loss at 163° C. and the very slight increase in float test of the residue as compared with the original material. While in consistency and percentage of asphaltenes and fixed carbon it is superior to the paraffin residuum, it would nevertheless make an unsatisfactory carpeting medium.

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The asphaltic residuum is from a California petroleum and, while greatly superior to the other two as a carpeting medium, would prove far from ideal unless used very sparingly so that the carpet would be very thin. It is quite viscous, as shown by its specific viscosity at  $100^{\circ}$  C. and its float test at  $50^{\circ}$  C. It also hardens up to some extent under the volatilization test, producing a residue of about the same consistency as that of the medium crude asphaltic petroleum. It is, however, preferable to the latter for use as a carpeting medium as in its original condition it represents a more advanced stage of hardening and of cementitiousness, and will not so readily emulsify with water and produce an oily mud on the road surface. A fairly thick bituminous carpet constructed with such a product is, however, more than apt to shove under traffic, owing to its mechanical instability, indicated by its relatively low percentages of asphaltenes and fixed carbon.

While none of the petroleum residuums have much to recommend them for direct use in road treatment or construction under ordinary conditions, they might all be satisfactorily used as fluxes for the harder asphalts in producing asphalt cements of various consistencies. They conform in characteristics to the three types of fluxes usually recognized as paraffin, semi-asphaltic and asphaltic. Fluxes produced from paraffin petroleum usually have a specific gravity of from 0.920 to 0.940. The semi-asphaltic fluxes lie between 0.940 and 0.975, while the asphaltic fluxes run from 0.975 to 1.010 and over. In general, as fluxes increase in specific gravity they must be used in greater quantity with a given refined asphalt to produce an asphalt cement of given penetration. Their flash point should be above  $163^{\circ}$  C. as this is the temperature to which they are likely to be heated when being combined with the solid asphalts. They should not lose over 5% by weight when subjected to the volatilization test for 5 hours at  $163^{\circ}$  C. The residues from this test may or may not harden appreciably, but the degree of hardening should never cause the asphalt cement in which they are incorporated to show a loss of over 50% in penetration when subjected to the volatilization test at  $163^{\circ}$  C.

Fluxes, will be again considered in connection with asphalts and asphalt cements.

The maltha is of California origin and possesses certain characteristics of a cut-back asphalt. Although a crude product, it is very viscous, as shown by its viscosity at  $100^{\circ}$  C., and is susceptible only to hot application. Considerable care should be exercised in heating it, owing to its low flash point. It shows a relatively high loss at  $163^{\circ}$  C. The fact that the residue gives a high float test at  $50^{\circ}$  C. and that it is truly an asphalt cement, as indicated by its penetration, shows an absence of non-volatile oils. The material is therefore similar to a soft asphalt cement cut-back with light volatile oils. As a carpeting medium it is greatly superior to any other product in the table so far considered. After application it should rapidly harden to a point where with a dressing of stone chips it would form a relatively stable carpet which should not shove under traffic if it were not over one-half inch in thickness. Its high percentage of asphaltenes and relatively high percentage of fixed carbon for a crude fluid product indicate its highly asphaltic nature. If topped it would in many respects resemble the cut-back asphalt shown in the last column.

The cut-back asphalt is almost a semisolid, as shown by its float test at  $50^{\circ}$  C. It has a higher specific gravity than any of the other products. While its flash point is relatively high and its loss at  $163^{\circ}$  C. small, the residue is a relatively hard asphalt cement having a penetration of 98. Its high percentage of asphaltenes and fixed carbon indicates its asphaltic nature. This material might make an even better carpeting medium than the maltha were it not for the fact that it is so nearly semisolid that great difficulty would be experienced in getting it to stick to an ordinary road surface even if first heated to a very fluid state. It is, however, admirably suited to the surface treatment of a bituminous macadam or bituminous concrete surface to replace an old seal coat after it has practically worn away. Such materials have been used to some extent in the construction of bituminous concrete roads. When heated they possess the advantage of being able to mix



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with and thoroughly coat coarse mineral aggregates, but until they have lost the light oils used in cutting-back the asphalt cement they do not possess sufficient mechanical stability to hold the aggregate in place under heavy traffic. When not exposed to surface conditions they do not lose their volatiles for a very long time and the road is therefore apt to shove and wave under traffic.

#### INTERPRETATION OF RESULTS IN TABLE II

In Table II, on page 128, are given the characteristics of certain emulsifying oils and emulsions. No general scheme of examination in common use is given for these products as they are not so widely used as the other fluid products, and the exact method of examination should depend upon their prospective use and just what characteristics are developed as the analyst proceeds in his examination.

The emulsifying oil consists mainly of what is known as an alkaline sludge obtained by refining certain petroleum distillates with caustic soda. While it does not contain water, it may be mixed or diluted with water to almost any desired degree of fluidity and therefore readily applied at any desired rate to a road surface. The character of the residue from the volatilization test and the low percentage of asphaltenes and fixed carbon show it to be unsuitable for any use other than as a dust preventive.

The residual emulsion contains a large amount of water, which is mostly driven off in the volatilization test at 105° C., leaving a viscous, sticky petroleum residue, together with any non-volatile soaps or soap-forming ingredients. Such a material may be diluted with water and applied to a road surface at any desired rate. It is primarily of value as a dust preventive, but successive applications tend to fill up the interstices of the road surface with a petroleum residuum which in very thin layers hardens and will not again emulsify with water. The asphaltic character of the material is shown by its percentage of asphaltenes and fixed carbon.



The other two emulsions carry even a higher percentage of water than the residual emulsion because they have been produced from asphalt cements as shown by the penetrations of

TABLE II  
ANALYSES OF EMULSIFYING OILS AND EMULSIONS

Test	Emulsifying Oil	Residual Emulsion	Asphalt-Cement Emulsion	Fluxed Native Asph. Emulsion
Sp. Gr. 25°/25° C.....	0.976	0.962	0.973	1.038
Sp. Vis., Engler, 50 c.c., 50° C.....	23.6	.....	.....	.....
Flash Point, open cup..	173° C.	.....	.....	.....
Loss, 105° C., 5 hrs., 50 grams.....	.....	19.9%	24.76%	52.5%
Pen. Res., 25° C., 100 grams, 5 sec.....	.....	Viscous Sticky	221	195
Loss, 163° C., 5 hrs., 50 grams (additional)...	14.8%	.....	.....	3.4%
Pen. Res., 25° C., 100 g., 5 sec.....	Fluid	.....	.....	72
Total Bitumen (Sol. in CS <sub>2</sub> ).....	96.42%	Tests on Residue 99.08%	Tests on Residue 96.90%	.....
Organic matter insoluble.....	0.22	0.81	1.90	.....
Ash as alkaline carbonate.....	3.36	0.11	1.20	.....
	100.00%	100.00%	100.00%	
Bit. insol. 86° B. naph. (Asphaltenes).....	1.2%	11.83%	19.9%	.....
Fixed Carbon.....	0.6%	4.28%	10.2%	.....
Approx. Composition:				
Water.....	.....	21.41	.....	49.50
Ammonium (NH <sub>3</sub> )...	.....	.....	.....	0.35
Fatty and Resin Acids	.....	.....	.....	9.45
Total Bitumen.....	.....	.....	.....	36.30
Organic matter insol., CS <sub>2</sub> .....	.....	.....	.....	1.10
Inorganic matter....	.....	.....	.....	3.30
				100.00

the residues obtained from the volatilization test at 105° C. Such products have been used in the construction of cold-mixed bituminous concrete pavements, but when so used a very considerable period of time is required for the water to evaporate sufficiently to develop the necessary consistency of bituminous



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cement which such highways demand. Their greatest value would appear to lie in their use for cold mixes to be used in patching bituminous roads. When so used the mix hardens much more rapidly than when laid in large masses as in construction work.

## SEMISOLID AND SOLID PETROLEUM AND ASPHALT PRODUCTS

Semisolid petroleum and asphalt products are used as asphalt cements or joint fillers. The solid products are usually fluxed with petroleum residuums to produce asphalt cements prior to use.

Asphalt cements intended for use in bituminous macadam construction are generally softer than for bituminous concrete or sheet asphalt pavements. As a rule the finer the mineral aggregate which is to be cemented the lower should be the penetration of the asphalt cement. This is due to the fact that compacted fine aggregate possesses less resistance to displacement in a pavement than coarse aggregate, and therefore requires a more solid and harder cement to hold the mineral particles in place. The most desirable penetration for asphalt cements for a given type of pavement varies with the type of oil or native asphalt from which they are produced, and the traffic and climatic conditions to which the pavement will be subjected. Asphalt fillers unless especially prepared by the addition of fine mineral matter are nothing more than asphalt cements particularly adapted to waterproof joints as the pavement expands and contracts. All of these products are ordinarily subjected to the following tests for the purpose of identification, control, and determination of their suitability for a specific purpose.

### ORDINARY TESTS

1. Specific Gravity  $25^{\circ}/25^{\circ}$  C.
2. Penetration Test:
  - a. Always at  $25^{\circ}$  C., 100 grams, 5 seconds.



- b. Frequently at  $0^{\circ}$  C. or  $4^{\circ}$  C., 200 grams, 1 minute, when used in cold climates.
- c. Sometimes at  $46^{\circ}$  C., 50 grams, 5 seconds, when used with fine aggregates or as fillers.
- 3. Melting Point  $^{\circ}$  C. for the harder varieties.
- 4. Ductility at  $25^{\circ}$  C. sometimes for the harder varieties.
- 5. Flash Point  $^{\circ}$  C. (usually open cup).
- 6. Volatilization Test at  $163^{\circ}$  C., 50 grams, 5 hours.
- 7. Penetration of Residue from Volatilization Test, penetration at  $25^{\circ}$  C., 100 grams, 5 seconds.
- 8. Total Bitumen:
  - Organic Matter Insoluble in Carbon Disulphide.
  - Inorganic Matter Insoluble in Carbon Disulphide.
- 9. Asphaltenes.
- 10. Carbenes.
- 11. Fixed Carbon.

## INTERPRETATION OF RESULTS IN TABLE III

Table III, on page 131, shows the results of these tests upon typical oil asphalts or asphalt cements and fillers. In comparing test values it should be remembered, however, that the various characteristics of the asphalt cements are the result not only of the type of oil distilled but also of the exact method of distillation.

Considering first the three Mexican oil asphalts, it will be noticed that as the penetration at  $25^{\circ}$  C. decreases the specific gravity, melting point, flash point, asphaltenes, and fixed carbon increase. On the other hand, the penetration at  $0^{\circ}$  C., the ductility, the loss at  $163^{\circ}$  C., and penetration of the residue decrease. The solubility in carbon disulphide and carbon tetrachloride, however, remain about the same as they are all practically pure bitumen and have not been subjected to injurious cracking. Their penetration at  $25^{\circ}$  C. would represent their relative suitability under favorable conditions for use as bituminous cements in the construction of bituminous macadam pavements, coarse aggregate bituminous concrete, and sheet

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TABLE III  
ANALYSES OF OIL ASPHALTS AND FILLERS

Test	MEXICAN			California	Texas	Blown Filler
	(1)	(2)	(3)			
Sp. Gr. 25°/25° C.....	1.034	1.041	1.046	1.035	1.021	0.999
Penetration 25° C., 100 g., 5 sec.....	125	90	55	85	95	45
Penetration 0° C., 200 g., 1 min.....	.....	42	18	14	45	27
Penetration 46° C., 50 g., 5 sec.....	.....	.....	250	.....	.....	67
Melting Point (cube).....	40° C.	50° C.	57° C.	48° C.	56° C.	82° C.
Ductility 25° C., cm.....	100+	85	60	100+	40	4
Flash Point (open cup).....	220° C.	240° C.	250° C.	205° C. +	205° C. +	205° C. +
Loss 163° C., 50 grams, 5 hours.....	1.8%	0.9%	0.3%	1.0%	0.4%	0.3%
Pen. Res., 25° C., 100 g., 5 sec.....	90	60	35	45	70	35
Total Bitumen (sol. in CS <sub>2</sub> ).....	99.90%	99.88%	99.85%	99.92%	99.85%	99.88%
Organic Matter insol.....	0.05	0.06	0.07	0.04	0.08	0.10
Inorganic Matter insol.....	0.05	0.06	0.08	0.04	0.07	0.02
	100.00%	100.00%	100.00%	100.00%	100.00%	100.00%
Bit. insol. 86° B. naph. (Asphaltenes).....	23.2%	26.3%	28.1%	21.3%	24.8%	31.2%
Carbenes.....	0.0%	0.0%	0.0%	0.02%	0.07%	0.08%
Fixed Carbon.....	13.2%	15.1%	16.5%	11.8%	13.0%	10.1%

asphalt topping. A somewhat higher penetration than that shown by No. 3 would be advisable for use in the binder course of a sheet asphalt pavement.

The relative characteristics of oil asphalts from different types of petroleum are illustrated by a comparison of the Mexican No. 2, the California, and the Texas, all of which have approximately the same penetration at 25° C. These products show evidence of having been manufactured primarily by careful steam distillation. It will be noted that for approximately the same consistency there is a general decrease in specific gravity from the Mexican to the Texas asphalt. The California asphalt is more susceptible to temperature changes than are the other two as shown by its relative penetration at 25° C. and 0° C. It is therefore naturally the most ductile. It also shows the lowest per cent of asphaltenes and fixed carbon, which is characteristic. The Mexican asphalts show a typically high percentage of fixed carbon. The flash points of all three products are over 205° C. and their low loss at 163° C., together with the penetration of their residues, well above 50% of the original materials, show that they will not unduly harden during their manipulation in construction or under service conditions.

The filler which is produced by blowing a heavy asphaltic residuum shows a much lower specific gravity than do any of the other materials, although its penetration is the lowest. It has a higher melting point and is much less susceptible to temperature changes, as shown by its relative penetration at three temperatures, than any of the others. Its ductility and percentage of fixed carbon are low, this being characteristic of all highly blown petroleum. Its percentage of asphaltenes is characteristically high. Its flash point, loss at 163° C., and the relative penetration of its residue are similar to the other asphalts. As a filler it is much more satisfactory than the other materials for, existing in a practically pure state in the pavement joints, it will not become unduly hard in cold weather nor bleed in hot weather. The blowing process may be used to impart to a petroleum residuum certain desirable charac-



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teristics, from the standpoint of a particular use, which cannot be secured in any other way. Besides being particularly adapted for use as fillers, blown or partly blown asphalts of the type shown are admirably adapted for use as seal-coating mediums on certain types of coarse aggregate bituminous concrete pavements. If of suitable consistency their peculiar rubbery nature makes them more permanent surfacing materials than the straight distilled products.

#### INTERPRETATION OF RESULTS IN TABLE IV

Table IV, on page 134, shows the results of tests upon typical refined native asphalts of most importance at the present time and of certain asphalt cements produced by fluxing them with residual petroleums or fluxes. It should be remembered that the characteristics of asphalt cements produced from any given native asphalt may be largely controlled not only by the amount of flux but also by the type of flux used. As the refined native asphalts are seldom if ever used without modification in highway work, it is seldom considered necessary to make or report all tests to which the asphalt cements are ordinarily subjected.

Comparing first the unfluxed materials it will be seen that the refined Trinidad and Cuban asphalts have by far the highest specific gravities. This is due to their very high percentage of mineral or inorganic matter insoluble in carbon disulphide. Refined Trinidad lake asphalt is very uniform in character, while Cuban asphalt varies considerably, according to the deposit from which it is taken. Refined Bermudez asphalt has a much lower specific gravity and percentage of mineral matter than the Trinidad and Cuban, while gilsonite, which is practically pure bitumen, has the lowest specific gravity of all. The refined Bermudez has the highest penetration at 25° C., the others being much harder. As would therefore be expected, the Bermudez shows the lowest percentage of asphaltenes. With the exception of Bermudez, which is characteristically high for a hard R. A., the refined native asphalts show a low loss by volatilization at 163° C. The percentage of bitumen

TABLE IV  
ANALYSES OF NATIVE ASPHALTS AND ASPHALT CEMENTS

Test	TRINIDAD		BERMUDEZ			Cuban Refined	Gilsomite Native
	Refined	Fluxed	Refined	Fluxed			
				(1)	(2)		
Sp. Gr. 25°/25° C.....	1.400	1.210	1.070	1.066	1.045	1.305	1.040
Penetration 25° C., 100 g., 5 sec.	7	50	25	48	130	0	0
Melting Point (cube).....	.....	55° C.	.....	59° C.	44° C.	.....	163° C.
Ductility 25° C., cm.....	.....	43	.....	30	.....	.....	.....
Flash Point (open cup).....	.....	205° C. +	.....	183° C.	192° C	.....	.....
Loss 163° C., 50 grams, 5 hrs...	1.0%	1.3%	4.0%	3.4%	3.1%	0.9%	0.4%
Pen. Res., 25° C., 100 g., 5 sec	.....	33	.....	18	66	0	0
Total Bitumen (sol. in CS <sub>2</sub> )....	56.53%	72.51%	95.00%	95.48%	96.89%	75.1%	99.77%
Organic Matter insol.....	6.97	4.32	2.05	1.77	0.93	3.5	0.04
Inorganic Matter insol.....	36.50	23.17	2.95	2.75	2.18	21.4	0.19
	100.00%	100.00%	100.00%	100.00%	100.00%	100.00%	100.00%
Bit. insol. 86° B. naph. (Asphal-							
tenes).....	36.9%	28.4%	32.1%	23.6%	21.8%	56.7%	53.7%
Carbenes.....	0.0%	0.0%	0.4%	0.3%	0.2%	0.0%	0.0%
Fixed Carbon.....	10.8%	11.2%	13.5%	12.0%	11.3%	25.0%	13.7%



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varies from almost 100 per cent in the case of gilsonite to as low as 56.5% in the case of Trinidad. The relative percentages of organic and inorganic matter insoluble in carbon disulphide, together with the character of such matter, helps to identify these materials. The ash of Trinidad asphalt, particularly the finer portion, is characteristically flesh pink, while the others are brown or reddish brown. The insoluble organic matter of Bermudez differs from the others in that it consists largely of residues of vegetation, such as very fine twigs, veins of leaves or grass, etc. Gilsonite has practically no insoluble organic matter. Little or no carbenes are present in any of the native asphalts and, in fact, both the Trinidad and Cuban are usually somewhat more soluble in carbon tetrachloride than in carbon disulphide. The percentage of fixed carbon varies within comparatively narrow limits with the exception of the Cuban, which is characteristically high.

As compared with the refined asphalts from which they are produced, the asphalt cements or fluxed asphalts in general show a decided decrease in specific gravity and asphaltenes with an increase in penetration and percentage of total bitumen. This is what would be expected from the fact that the flux is usually a viscous fluid residual petroleum consisting of practically pure bitumen with a lower specific gravity and percentage of asphaltenes than the refined asphalt. As specific gravity is usually an additive property in mixtures of bituminous materials if the specific gravity of the original R. A. and flux, together with that of the A. C., are known, the approximate properties of R. A. and flux may be calculated. The same is true of the percentage of bitumen. If, therefore, a refined asphalt is uniform in character it may be possible to calculate from the total bitumen of its A. C. alone how much flux has been used. In such case the flux is presumed to be pure bitumen.

The penetration of a fluxed asphalt will necessarily depend to a large extent upon the type of flux and the amount used. The more of a given flux which is combined with an asphalt the higher becomes the penetration of the asphalt cement. Also, in general, the heavier the flux the more is required to



produce an asphalt cement of given penetration from a given asphalt. Light fluxes prepared from a given petroleum as a rule produce asphalt cements less susceptible to temperature changes than do the heavier fluxes produced from the same petroleum, but they are apt to show a higher loss by volatilization. Fluxes which are suitable for one asphalt will not always produce satisfactory asphalt cements from another asphalt. It is therefore frequently advisable to study various combina-

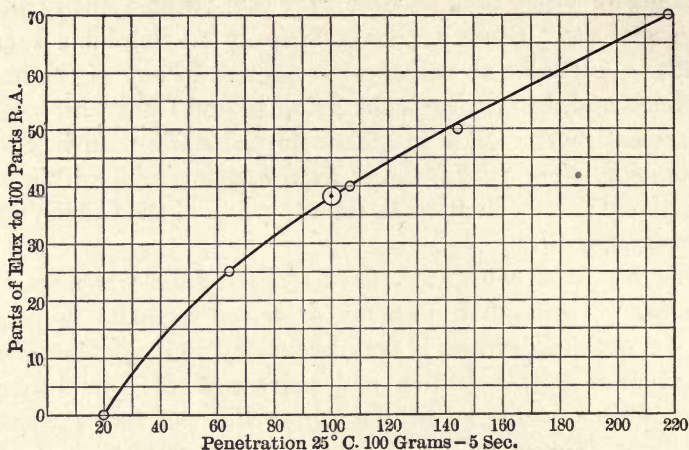


FIG. 39. Example of Fluxing Curve

tions in the laboratory before making up large batches of asphalt cement in the plant or factory.

While experience will often indicate the proportion of a given flux to add to a refined asphalt in order to produce an asphalt cement of any desired penetration, it is often desirable to prepare a fluxing curve in the laboratory. This is done by making up a number of trial mixes of known proportions and plotting the penetrations of the resulting asphalt cements against the parts of flux used to 100 parts of the refined asphalt, as shown in Fig. 39. If these mixtures have been properly selected it will then be possible to construct from these plotted points a curve from which may be obtained with reasonable accuracy the amount of the given flux to use if it is desired to secure an asphalt cement of a given consistency.

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The value of this method at once becomes apparent when it is realized that to produce an asphalt cement of 50 penetration from refined Trinidad asphalt may usually require from 20 to 60 parts of flux per hundred, while in the case of refined Bermudez from 9 to 30 parts only may be required to produce an asphalt cement of the same penetration.

No matter how large a proportion is used, a good flux should not materially raise the loss at  $163^{\circ}$  C. of an asphalt nor should it tend to harden to such an extent as to cause the residue of the asphalt cement to exhibit an undue decrease in penetration. Of the asphalt cements shown in the table it will be noticed that in the case of Bermudez the loss by volatilization has even been decreased by the addition of flux. Like the oil asphalts, fluxed native asphalts of various consistencies are required for the different types of construction.

Cuban asphalts and gilsonite are less used in the United States for road and paving purposes than are Trinidad and Bermudez asphalts. Gilsonite, has, however, to a considerable extent been combined with relatively large quantities of blown petroleum residuums to form what are known as blown-oil gilsonite products, which are cementitious and possess a very marked rubbery characteristic.

## REFINED TARs AND TAR PITCHES

Refined tars are used as dust preventives, carpeting mediums, or bituminous cements, largely according to their consistency. The bituminous cements are usually soft pitches, while the harder tar pitches are sometimes used as joint fillers.

Crude tars are sometimes used as dust preventives. Most coal tars are, however, too viscous for successful cold application and have to be combined with distillates or very fluid tars in order to make them applicable for such work. Water-gas tars and some coke-oven tars may, however, be applied cold in the crude or partially dehydrated state. High carbon tars are totally unsuited for use as dust preventives, and the

majority of the dust preventives are, therefore, generally manufactured from low carbon crudes. In order to so use tars containing as high as 5 per cent of free carbon, it is not unusual to have them combined with a small amount of water, which increases their fluidity or, in other words, decreases their viscosity. Partly dehydrated coke-oven tars combined with water-gas tars may be made to serve this purpose. Owing to the fact that the dust preventives tend to volatilize and harden more rapidly than petroleum dust preventives, their excessive use is not so apt to disintegrate the road through lubrication. Their repeated use may, moreover, result in the formation of a thin bituminous carpet.

Tar carpeting mediums are usually straight distilled residual tars which are too viscous to apply cold. They ordinarily possess more cementitiousness than a petroleum product of similar viscosity and harden more rapidly after application to a road surface. If used in excess, however, they are apt to produce with the covering of mineral matter an unstable and wavy carpet. Their initial hardening, which is a desirable feature, is, under ordinary conditions, apt to continue until the carpet becomes so hard that it wears more rapidly under traffic than does the petroleum carpet. This is particularly true of those materials which contain a high percentage of the lighter oils or naphthalene and free carbon. For carpet work this general tendency to harden even too much is preferable to not hardening sufficiently. In some cases petroleum or asphalt products are combined in relatively small proportions with tar carpeting mediums, in order to reduce the tendency to harden.

Because of their tendency to harden after use and their susceptibility to temperature changes, the refined tars used in bituminous macadam and bituminous concrete pavements are usually much softer than either the oil asphalts or fluxed native asphalts used in the same type of construction. This is admissible when the aggregate is coarse, because of the high cementitiousness of the soft semisolid tars residues. For fine aggregates where mechanical stability is largely dependent upon the hardness of the bituminous cement, tars are not generally used.



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For the coarser aggregate pavements the same grade of tar used in the construction of the wearing course proper is also commonly used as a seal coat. Although such a seal coat is apt to harden more rapidly, perhaps, than desirable, in the case of the bituminous macadam the underlying thick films of tar tend to enrich and prolong the life of the seal coat. In bituminous concrete construction, however, there is not the same excess of bitumen in the underlying course, and for this reason an asphalt seal coat is sometimes employed where a refined tar is used in the concrete proper.

Pitch fillers are manufactured from tars by distillation and sometimes blowing the tar residue to the desired melting point. They are often quite hard and brittle and carry to advantage a high percentage of free carbon. They are much more susceptible to temperature changes than are the blown petroleum or asphalt products prepared for the same purpose.

The refined tars and tar pitches are ordinarily subjected to the following tests in routine examination:

## ORDINARY TESTS

1. Specific Gravity  $25^{\circ}/25^{\circ}$  C.
2. Specific Viscosity (Engler) 50 cubic centimeters at  $40^{\circ}$  or  $50^{\circ}$  C. for materials for cold surface application.
3. Float Test at  $50^{\circ}$  C. for materials which have to be heated, except the hard tar pitches.
4. Penetration Test at  $25^{\circ}$  C., 100 grams, 5 seconds, for hard tar pitches.
5. Melting Point  $^{\circ}$  C., cube method, for the harder tar pitches.
6. Distillation Test (flask method):
  - Water.
  - Distillate to  $110^{\circ}$  C.
  - Distillate  $110^{\circ}$ – $170^{\circ}$  C.
  - Distillate  $170^{\circ}$ – $270^{\circ}$  C.
  - Distillate  $270^{\circ}$ – $300^{\circ}$  C.
  - Pitch Residue.
7. Melting Point  $^{\circ}$  C. (cube method) of residue from distillation test.



## 8. Total Bitumen:

Organic Matter Insoluble in Carbon Disulphide (free carbon).

Inorganic Matter Insoluble in Carbon Disulphide.

## 9. Dimethyl sulphate test when the presence of petroleum or asphalt products is required or suspected.

## INTERPRETATION OF RESULTS IN TABLE V

Table V, on page 141, shows the results of these tests upon typical tar products prepared for use in highway work

In comparing these analyses it will first be noticed that for the same purpose refined water-gas tars show a lower specific gravity and a lower percentage of free carbon than do the others. If the three water-gas tars are considered it will also be seen that their specific gravities increase as they become more viscous and show a higher float test.

The first analysis is that of a dust preventive produced from a water-gas tar by distillation of the more volatile constituents. The second is one produced from a low carbon coal tar and shows the presence of both water and light oils. The water is largely responsible for the low specific viscosity of this material as compared with the first sample. Its specific gravity and percentage of free carbon indicate a coke-oven tar. Both products show practically the same amount of pitch residue. It has been found that in general refined tars with as high as 30 specific viscosity at 50° C. may be successfully applied cold. Both products are therefore suitable for cold application.

The first carpeting medium is a tar asphalt compound as evidenced by the positive result of the dimethyl sulphate test. Its specific gravity and very low percentage of free carbon show it to be largely a refined water-gas tar. The second product contains no petroleum or asphalt as shown by the negative result from the dimethyl sulphate test. Its percentage of free carbon comes just within the maximum limit for what is considered the best type of tar carpeting medium. Its float test at 50° C. is lower than for the water-gas tar, although it has a higher percentage of pitch residue. This is due to the fact



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TABLE V  
ANALYSES OF REFINED TARs

Tests	DUST PREVENTIVES		CARPETING MEDIUMS		BITUMINOUS CEMENTS		
	Water Gas	Coke Oven	Water Gas	Coke Oven	Water Gas	Coke Oven	Gas House
Sp. Gr. 25°/25° C. ....	1.118	1.149	1.158	1.210	1.172	1.215	1.258
Sp. Vis. (Engler) 50° C., 50 c.c. ....	23.7	10.9	85"	45"	158"	140"	170"
Float Test 50° C. ....	.....	.....	.....	.....	40° C.	42° C.	43° C.
Melting Point (cube) ....	.....	.....	.....	.....	.....	.....	.....
Distillation (flask method):							
Water, by volume....	0.0%	1.7%	0.0%	0.0%	0.0%	0.0%	0.0%
Distillate to 110° C., " ..	0.3	0.9	0.0	0.0	0.0	0.0	0.0
Distillate 110° to 170° C " ..	0.8	2.5	0.3	0.0	0.7	0.3	0.0
Distillate 170° to 270° C " ..	21.9	18.4	6.8	14.5	6.2	5.4	9.4
Distillate 270° to 300° C " ..	7.3	4.6	17.9	8.0	16.0	12.9	6.5
Pitch Residue.....	69.7	71.9	75.0	77.5	77.1	81.4	84.1
	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%	100.0%
Melt. Pt. Residue (cube).....	.....	.....	65° C.	74° C.	65° C.	71° C.	85° C.
Total Bitumen (sol. in CS <sub>2</sub> ).....	97.67%	95.05%	98.50%	85.05%	99.12%	86.60%	70.57%
Organic Matter insol. (free carbon).....	2.22	4.89	1.45	14.93	0.83	13.38	29.40
Inorganic Matter insol .....	0.11	0.06	0.05	0.02	0.05	0.02	0.03
	100.00%	100.00%	100.00%	100.00%	100.00%	100.00%	100.00%
Dimethyl Sulphate Test.....	.....	.....	Positive	Negative	.....	.....	.....



that the actual percentage of bitumen in its pitch residue is much lower and it therefore contains, on a bitumen basis, a much greater proportion of oils distilling below  $300^{\circ}$  C. While originally softer, it would therefore tend to harden more rapidly in service than the water-gas tar product. This is further indicated by the higher melting point of its pitch residue. As shown by their float test, both products possess a satisfactory original consistency for building up a bituminous carpet and would have to be applied in a heated state.

Refined tars used as cements in bituminous macadam or coarse aggregate bituminous concrete pavements usually show a float test at  $50^{\circ}$  C. of from 2 to 3 minutes. All of the last three products come within these limits. Although of similar consistency, it will be noted that the residual water-gas tar shows the lowest percentage of free carbon and the residual gas-house coal tar the highest. The specific gravity of these tars increases with the percentage of free carbon. All of the materials show a very low percentage of ash, and the percentage of total bitumen is therefore almost entirely dependent upon the amount of free carbon present. All of the residuals have been prepared at sufficiently high temperatures to remove the more volatile oils, as shown by the results of distillation. The relation of total distillate to pitch residue is found to vary somewhat but the ratio of total distillate to actual bitumen in the pitch residue will, upon calculation, be found to be nearly the same. In this connection it should be remembered that all of the free carbon concentrates in the residue during distillation. If, for example, the original crude tar contains 10 per cent of free carbon and the pitch residue amounts to 50 per cent of the crude, that residue would then consist of not less than 20 per cent or one-fifth free carbon.

Free carbon in tars exists in the form of finely divided amorphous black particles resembling soot or lamp-black. These particles are held in suspension in the tar and will not readily settle out even if the tar is very fluid. If filtered from the bitumen the free carbon is found to be a dry black powder entirely lacking in cementitiousness. Ultimate analysis indicates

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that it is not, however, pure carbon but probably a mixture of hydrocarbons very rich in carbon, which have been produced by an advanced stage of cracking during the original formation of the tar. For all practical purposes, however, it may be considered as carbon and as an inert impurity in the tar bitumen. It is non-volatile at ordinary distilling temperatures, and for this reason always remains in the residue of a tar distillation. As a very high percentage of free carbon is not considered desirable in refined tars commonly used in highway engineering, those crude tars which originally carry a high percentage cannot well be utilized in the manufacture of such products unless mixed in suitable proportions with a low carbon crude tar prior to distillation. If both low and high carbon tars are available to the tar refiner he is able by judicious mixing to obtain a combination which upon subsequent distillation will yield a refined tar containing less than the maximum limit of free carbon which has been set. Knowing the per cent of distillate which will have to be removed in order to produce a residue of desired consistency, the percentage of free carbon in the refined tar may be closely approximated from the percentage present in the material distilled. If, however, the tar is badly cracked or locally overheated during distillation, additional free carbon will be formed. As all tars originally contain some free carbon, however, it is impossible from its determination in the finished product to ascertain whether or not cracking has taken place during distillation as in the case of petroleum products. While chemically inert, free carbon has a decided effect upon certain physical characteristics of the tar in which it occurs. It increases the gravity of a tar and also its viscosity or apparent consistency to a marked extent. In a general comparison of low and high carbon tars the following facts are of interest to the highway engineer. For refined tars of the same degree of hardness those of low free carbon contents have greater inherent binding strength than those of high carbon contents. In refined tars whose bitumen is of the same degree of hardness those high in free carbon have a greater inherent binding strength than those low in free carbon, but

the binding capacity of the former is lower because of the lower percentage of bitumen present. The waterproofing value of high carbon tars is in general less than that of low carbon tars. A high percentage of free carbon tends to retard absorption of the tar bitumen by porous surfaces. When a tar is exposed in comparatively thin films free carbon has little or no effect in retarding volatilization of the lighter bituminous constituents. In certain classes of tar aggregates free carbon may serve as a filler and add to the mechanical strength of the aggregate, but finely divided mineral matter, which may be incorporated in any desired amount, is more satisfactory in this connection. In general it is considered desirable that a tar dust layer should not contain over 8% free carbon, that a tar carpeting medium should not contain over 15% free carbon, and that for bituminous macadam and bituminous concrete construction a refined tar should not contain over 20% free carbon. There is, however, some difference of opinion in this connection.

Naphthalene,  $C_{10}H_8$ , frequently occurs in tar in larger quantity than any other one hydrocarbon and for this reason it exerts an appreciable influence upon the physical and chemical properties of the tar. In the pure state it exists in white flaky crystals or scales melting at  $79^{\circ}C$ . and having a boiling point of  $218^{\circ}C$ . It has the characteristic odor commonly familiar in moth balls and is extremely volatile. It volatilizes far below its boiling point and from crude tars distills to a considerable extent with the aqueous vapors and also with the light tar oils. Even at ordinary temperatures it volatilizes slowly from both crude tars and refined tars in which it is present. No satisfactory method has as yet been devised for its quantitative determination in tars, but its presence in appreciable quantities is readily detected by its crystallization from certain fractions obtained by distilling the tar. In some tars it occurs to such a large extent that almost the entire distillate solidifies or crystallizes upon cooling. In the manufacture of straight residual refined tars for use in highway engineering some of the naphthalene is removed during distillation. A considerable proportion, however, usually remains behind in the residue. The pres-



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ence of this naphthalene exerts a marked influence upon the consistency of the tar and, as it is a volatile constituent, its effect in this connection is of considerable interest. From its nature it cannot be considered as a binding constituent, but, although it is solid, it may combine with and serve as a flux for those hydrocarbons which are directly responsible for the cementitiousness of tars. Thus by heating together a quantity of naphthalene and a hard tar pitch it is often possible to produce a soft and almost fluid product. In this connection it has been found that the fluxing value of naphthalene for hard tar pitches is somewhat greater, although quite similar to the heavier tar distillates free from naphthalene or other crystallizable solids. This is true until the mixture becomes so saturated as to cause the naphthalene to precipitate. For the harder tar pitches the addition of very small percentages of naphthalene produces a more marked increase in fluidity than for originally softer pitches. On the other hand where naphthalene is present beyond its point of saturation it decreases the fluidity of the product at temperatures below its melting point, but at higher temperatures it continues to increase the fluidity of the product. The conclusions to be drawn from the above facts are that refined tars containing a high percentage of naphthalene, although the lighter tar oils may be absent, may be expected to harden rapidly upon exposure through loss of the naphthalene by volatilization. This is particularly true when the refined tar is used for surface treatment.

## CREOSOTING OILS OR WOOD PRESERVATIVES

There is probably no class of bituminous materials over which there is a wider diversity of opinion relative to desirable characteristics than creosoting oil or wood preservatives. The oldest and apparently the most favored type are the coal-tar distillates. There are, however, a growing number of advocates of water-gas tar oils and of fluid refined tars or mixtures of refined tars with tar distillates.

## ORDINARY TESTS

The wood preservatives are now commonly subjected to the following tests:

1. Specific Gravity  $38^{\circ}/38^{\circ}$  C.
2. Specific Viscosity (Engler)  $82^{\circ}$  C.
3. Material insoluble in Benzol and Chloroform.
4. Water.
5. Distillation Test (Retort Method):

Distillate  $200^{\circ}$ – $210^{\circ}$  C.

“  $210^{\circ}$ – $235^{\circ}$  C.

“  $235^{\circ}$ – $315^{\circ}$  C.

“  $315^{\circ}$ – $355^{\circ}$  C.

Residue (Character).

6. Specific Gravity  $38^{\circ}$  C. of Distillate from  $235^{\circ}$  to  $315^{\circ}$  C.
7. Specific Gravity  $38^{\circ}$  C. of Distillate from  $315^{\circ}$  to  $355^{\circ}$  C.

An idea of the two most common types of wood preservatives may perhaps best be obtained from Table VI, which is an abstract of two specifications published in the 1916 report of Committee D-7 of The American Society for Testing Materials.

TABLE VI

## SPECIFICATION LIMITS FOR WOOD PRESERVATIVES

Test	Coal-Tar Oil	Water-Gas Tar Oil
Sp. Gr. $38^{\circ}/38^{\circ}$ C.....	1.06–1.12	1.11–1.14
Sp. Vis. (Engler) $82^{\circ}$ C.....	Not over 1.3	.....
Material insol. hot benzol, chloro- form.....	Not over 3.0%	Not over 2.0%
Water.....	Not over 3.0%	.....
Distillation (Retort Method)		
Up to $210^{\circ}$ C., by weight.....	Not over 5.0%	Not over 3.0%
Up to $235^{\circ}$ C., by weight.....	Not over 30.0%	Not over 10.0%
Up to $315^{\circ}$ C., by weight.....	35.0–70.0%	Not over 40.0%
Up to $355^{\circ}$ C., by weight.....	Not under 65.0%	Not under 25.0%
Sp. Gr. $38^{\circ}$ C. of Distillate from $235^{\circ}$ to $315^{\circ}$ C.....	Not under 1.02	0.96–1.00
Sp. Gr. $38^{\circ}$ C. of Distillate from $315^{\circ}$ to $355^{\circ}$ C.....	Not under 1.08	.....



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## INTERPRETATION OF RESULTS IN TABLE VI

In comparing these test values it will be noted that while the specific gravity of the water-gas tar oil is allowably higher than the coal-tar oil, the specific gravity of its distillate from  $235^{\circ}$  to  $315^{\circ}$  C. is much lower. This practically prevents a mixture of appreciable quantities of one type with another. The specific gravity of the water-gas tar oil practically necessitates the presence of a considerable quantity of pitch while that of the coal-tar oil requires a large amount of coal-tar distillate and allows for a relatively small amount of coke-oven tar pitch or an exceedingly small proportion of gas-house tar pitch. The latter is further restricted by the 3.0% allowance for insoluble material. In neither case does the allowance for insoluble material reach a percentage which would seriously interfere with the impregnation of wood block through undue clogging of the cells. The distillation limits in general indicate that the coal-tar oil is more volatile and would therefore tend to evaporate more rapidly from the block. On the other hand it would produce a cleaner and less sticky block owing to its lower percentage of pitch. The relative value of oils covered by these specifications for waterproofing and preserving wood-paving block and producing a satisfactory pavement would depend both upon their manipulation during the impregnating process and upon conditions to which the finished pavement would be subjected.

## BITUMINOUS AGGREGATES

Bituminous aggregates are ordinarily examined only for the percentage of bitumen and the mechanical analysis or grading of the mineral aggregate which may either be reported upon a percentage basis of the original mix or upon the mineral matter alone. In some cases it is advisable to recover the bitumen for the purpose of identification and frequently for its specific gravity. The specific gravity of the mineral matter may also be determined to advantage.



## RATIONAL PER CENT OF BITUMEN

The determination of the percentage of bitumen as ordinarily made is upon a weight basis. As a means of comparing different bituminous aggregates the weight basis is irrational unless the relations of weight to volume are the same for the mixtures compared. In any bituminous aggregate the volume of bitumen is a most important factor from the standpoint of covering capacity, thickness of film, and, in some cases, the reduction of voids. In view of the rather wide variation in specific gravity of ordinary bituminous cements and mineral aggregates the volume method of comparison is the most rational. The volume or rational per cent of bitumen in a bituminous aggregate may be determined by recovery of the bitumen after its extraction and determination upon a weight basis. The specific gravity of the recovered bitumen should be determined as well as that of the mineral aggregate. From this data the volume proportions of each are ascertained by dividing their weight per cent by their respective specific gravity. These proportions transposed to total 100 are the rational or volume percentages of the constituents. The formula for expressing the rational per cent of bitumen is as follows, where  $x$  represents the rational per cent of bitumen,  $p$  the weight per cent of bitumen,  $a$  the specific gravity of the bitumen, and  $b$  the specific gravity of the aggregate:

$$x = \frac{100 \, pb}{pb + a(100 - p)}$$

The importance of the use of the rational per cent of bitumen as a basis of comparison may be illustrated by considering two bituminous aggregates which contain 6% by weight of bitumen and are practically identical in so far as the consistency of bitumen and grading of the aggregates are concerned. According to the ordinary interpretation of results of analysis the two mixtures might be considered as equivalents. If, however, it is found that the first mix is composed of an aggregate with

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a specific gravity of 2.50 while the recovered bitumen shows a specific gravity of 1.17 and that the aggregate of the second mix has a specific gravity of 3.50 while the recovered bitumen shows a specific gravity of 0.960, the rational per cent of bitumen would be found to differ greatly in the two mixes, as follows:

<i>Constituents</i>	<i>% by Wt.</i>		<i>Sp. Gr.</i>		<i>Rational Proportion</i>		<i>Rational %</i>
<i>First mix:</i>							
Aggregate.....	94	÷	2.50	=	37.6	=	88.0
Bitumen.....	6	÷	1.17	=	5.1	=	12.0
	<hr/>						<hr/>
	100						100.00
<i>Second mix:</i>							
Aggregate.....	94	÷	3.50	=	26.9	=	81.0
Bitumen.....	6	÷	0.96	=	6.3	=	19.0
	<hr/>						<hr/>
	100						100.0

## DENSITY AND VOIDS

Without reference to grading or practical possible compression, the theoretical maximum density of a compressed bituminous aggregate may be calculated from the specific gravity and relative proportions of the individual constituents present. Where the individual constituents are at first hand this is a comparatively simple matter, but where a mixed aggregate or section of compressed pavement is under examination it is first necessary to remove and recover the bitumen, the percentage of which must also be determined. The specific gravity of recovered bitumen may then be determined and that of the mineral aggregate. If coarse particles are present it is advisable to screen the aggregate into two or more sizes and determine the specific gravity of each. After the gravity and per cent by weight of the various constituents have been determined, their volume proportions are calculated by dividing their weight percentages by their respective specific gravities. The ratio of the sum of the weight proportions to the sum of the volume proportions is then the theoretical maximum density of the mix. When the per cent by weight and specific gravity of two or three constituents are given, the following formulas may be used if  $D$  represents the maximum possible density,

$W$ ,  $W^1$  and  $W^2$  represent the per cents by weight and  $G$ ,  $G^1$  and  $G^2$  their respective specific gravities.

Two Constituents

$$D = \frac{100}{\frac{W}{G} + \frac{W^1}{G^1}}$$

Three Constituents

$$D = \frac{100}{\frac{W}{G} + \frac{W^1}{G^1} + \frac{W^2}{G^2}}$$

The probable maximum density of compressed bituminous aggregates may be determined experimentally in the laboratory by heating the mixed aggregate to proper working temperature and compressing it in a cylindrical mold fitted with a plunger which has also been heated to the same temperature. The plunger may be forced into the mold by means of a compression machine or heavy hammer. Upon cooling, the specimen is forced from the mold and its specific gravity determined by the ordinary displacement method. The probable maximum density is largely dependent upon the grading of the mineral particles, which is a most important consideration for fine aggregates. For a well-graded fine aggregate carrying the proper percentage of bituminous cement the probable maximum density should closely approach the theoretical maximum density.

The actual density of compressed bituminous aggregate, such as a section of a bituminous concrete pavement, is directly determined by means of the displacement method. If satisfactory compression has been obtained in the construction of the pavement, its density should closely approach the probable maximum density as obtained by laboratory test upon a section of the pavement which is first softened and disintegrated by warming and then compressed at proper working temperature as described in the preceding paragraph.

The per cent of voids in a compressed bituminous aggregate is determined from the actual density of the compressed aggregate as compared with the theoretical maximum density, which allows for no voids. Thus, if  $D$  represents the theoretical maximum density and  $d$  the actual density, the per cent of voids is determined by the following formula:

$$\% \text{ Voids} = \frac{100 (D - d)}{D}$$



## NOTES

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## NOTES

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If proper compression has been secured in the construction of a bituminous concrete pavement, the percentage of voids as above determined should closely correspond with the voids in a properly prepared laboratory compressed sample. If a fine aggregate is well graded and carries the proper percentage of bituminous cement, the voids should be very low and seldom over 3 or 4%.

## INTERPRETATION OF RESULTS IN TABLE VII

While it does not come within the scope of this manual to enter into an exhaustive consideration of the value of well-graded aggregates and the percentage of bitumen which they should properly carry, a few analyses of the more common types of bituminous aggregates are given in Table VII, on page 152, for the sake of comparison.

The sheet asphalt topping shown in this table is of excellent character and carries normal proportions of bitumen, filler, and sand. The sand grading is also good. For this type of aggregate it has been customary to adopt certain sand gradings as standard and then in practice to approach these standards as closely as possible. Examples of two such condensed standard gradings are as follows:

STANDARD SAND GRADINGS

	Heavy Traffic	Light Traffic
Passing 80 mesh and retained on 200 mesh....	34%	20%
Passing 40 mesh and retained on 80 mesh....	43%	45%
Passing 10 mesh and retained on 40 mesh....	23%	35%

The proper percentage of bitumen and filler will depend upon the exact grading. In general, the finer the mineral aggregate the greater will be the percentage of bitumen properly required to bind and waterproof it after compression.

From the above it will be seen that the bituminous sandstone shows a poorly graded aggregate relatively coarser than the topping and therefore requiring less bitumen. Such a prod-





## NOTES

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## NOTES

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uct after compression is much more readily shoved and disintegrated under traffic than is the topping, provided that in both cases the bitumen is of suitable consistency.

The asphalt block is composed of a compressed mixture of rock screenings, mineral filler, such as limestone dust, and bitumen. It is, in fact, a graded bituminous fine aggregate concrete. A graded coarse aggregate concrete would be obtained by using such a mix to fill the voids in a product such as shown in the last column.

The one-size broken-stone concrete is composed of a mixture of bitumen and one product of a stone-crushing and screening plant, commonly known as commercial  $\frac{3}{4}$ -inch broken stone. It is therefore ungraded. While carrying a rather high percentage of voids even after compaction on the road, this size stone, when suitably mixed with bitumen, possesses sufficient resistance to displacement to produce a satisfactory wearing surface under favorable conditions.











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